MECHANICAL AND SOLID PARTICLE EROSION BEHAVIOR OF MODIFIED RICE-HUSK FILLED EPOXY COMPOSITE

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE DEGREE OF

Master of Technology

In

Mechanical Engineering
(Specialization- Machine Design & Analysis)

By

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By
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Under the supervision of
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2011-2013

Dedicated to

My Parents

Mr. Baratu Bahadur Singh

&

Mrs. Uttara Devi

National Institute of Technology, Rourkela

769008, Orissa, INDIA

CERTIFICATE

This is to certify that the thesis entitled "**Mechanical and Solid Particle Erosion Behavior of Modified Rice-Husk Filled Epoxy Composite**" submitted to the National Institute of Technology, Rourkela (Deemed University) by **Ajeet Bahadur Singh, Roll No. 211ME1163** for the award of the Degree of Master of Technology in Mechanical Engineering (Specialization– Machine Design & Analysis) is a record of bonafide research work carried out by him under my supervision and guidance. The results presented in this thesis has not been, to the best of my knowledge, submitted to any other University or Institute for the award of any degree or diploma.

The thesis, in my opinion, has reached the standards fulfilling the requirement for the award of the degree of **Master of Technology** in accordance with regulations of the Institute.

Date: /June/2013

(Dr. S. K. Acharya)
Associate Professor
Mechanical Engineering Department

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ABSTRACT

In today's modern age growing population and product requirement has triggered the issue of low cost manufacturing material which should also be renewable resource to meet the requirement easily anywhere in the world. Composites are combination of two or more materials that provide characteristics which cannot be achieved by single material system. This is achieved by cohesion of the materials made by physically combining two or more compatible materials, different in characteristics, composition and sometimes in form. Efforts have been made in many researches in the field of composite to make it low cost, easy availability, ecofriendly and also that it can fulfill all the criteria of its ease of manufacturing and usage. Natural fibers have attracted attention of many researchers as filler for the composite materials because of their low cost, ecofriendly and ease of availability; hence natural fibers are suitable alternative of traditional synthetic and inorganic fibers. Accordingly extensive studies on preparation of polymer matrix composite (PMC) replacing the synthetic fibers with natural fibers like Jute, Sisal, Pineapple, Bamboo, Kenaf and Bagasse were carried out. These plant fibers have many advantages over glass fiber or carbon fiber like renewability, environmental friendly, low cost, lightweight and high specific mechanical performance.

Among the various biomasses, with abundant and renewable energy sources, rice husk (RH) is a potential source of energy which can easily be available anywhere since it is a byproduct of rice mill. It has already been utilized in many useful applications such as light weight concrete, an insulating material, fillers in plastics, building materials (bricks), panel boards and activated carbon, electricity generation and husk-fueled steam engines etc. Rice husk has a characterization of hard surface, high silicon content, small bulk density. Against this back ground the present research work has been undertaken with an objective to explore the use of natural fiber Rice-Husk, as a reinforcement material in epoxy base. Mechanical and solid particle erosion wear behaviors of Rice-Husk reinforced epoxy polymer composite have been studied. All these tests have been carried out as per ASTM standard. To study the mechanical and erosion properties of the composite, different volume fraction of fiber have been taken (5%, 10%, 15% & 20%). These fibers were randomly distributed in the matrix. Usual hand lay -up technique has

been adopted for manufacturing the composite. To have a good compatibility between the fiber and matrix, fibers were chemically treated with acetone and alkali. It was observed that tensile and flexural strength increases with increase in fiber content and best result was found for 15% wt. fraction of RH fiber reinforced epoxy composite. Solid particle erosion test was also carried out and significant reduction in erosion strength was found in the RH fiber filled epoxy composite compared to neat epoxy. It was observed that modified fiber increases the strength as well as erosion behavior appreciably. Erosion test of the samples characterized the RH-epoxy composite as a semi-brittle material. Finally scanning electron microscopy (SEM) and x-ray diffraction (XRD) tests has been carried out which further explains the characteristics of the composite samples.

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FRP	Fiber Reinforced Plastic
PMC	Polymer Matrix Composite
RH	Rice Husk
σ	Tensile Strength
δ	Deflection of the speciment
P	Load
A	Cross sectional Area
L	Span length
E	Young's modulus
UTM	Ultimate testing machine
FS	Flexural Strength
b	Breadth of specimen
t	Thickness of specimen
G	Flexural Modulus
y	Extension of the specimen
PRH	Plain Rice-Husk
E_r	Erosion Rate
v	Velocity of impingement
n	Velocity exponent
Δw	Weight loss
w_e	Weight of eroding particle
η	Erosion efficiency
H	Hardness of the composite
ρ	Density of the composite

Chapter-1

1.1 BACKGROUND

Composite materials have been used by men since ancient age and yarn is probably one of the greatest developments of mankind which enabled him to survive any climate area and to explore the surface of the earth hence flexible fabrics made of cotton, flax and jute were excellent compared to animal skins. Age after age use of the composite materials with natural resources increased continuously in the form of straw reinforced walls, composite bows and cross bows, chariots made of the combination of layers of wood, bones and horns. In today's modern age growing population and product requirement has triggered the issue of low cost manufacturing material which should possess mechanical properties good enough to meet the standards and which can also be renewable resource to meet the requirement easily anywhere in the world. Composite materials with natural fiber have attracted attentions of many researchers worldwide because of their low cost and ease of manufacturing.

In these days polymer composite materials are extensively used in engineering applications due to their excellent specific physical and chemical properties. They also find applications in fields where high resistance to wear, abrasion and erosion is required (mining, automobile, domestic equipment, aerospace, marine, sports etc.). Automobile industries are showing the highest interest towards natural fiber composites and also in many European countries they have already decided to use mostly bio-degradable and ecofriendly materials for the manufacturing of the automobile components especially in cars.

Composite materials have been seen in nature from small plants to huge trees they are all made of natural fiber composite with combination of cellulose-lignin compound with the hydrogen bonding, on the other hand we also see composite materials used by nature as teeth, bones & muscle tissues. The market research firm Frost & Sullivan says that, as early as 2015, lignin will be used commercially as replacement for phenol for manufacturing of resins, adhesives, surfactants or polyester. The advantage of natural fibers is their high stiffness, better strength to weight ratio and low cost whereas their disadvantage is they are highly sensitive to

moisture absorption. The moisture absorption sensitivity of natural fibers can be reduced by chemical processing of fibers but that induces extra cost but still we can afford a cost effective product compared to synthetic fiber composite and other materials.

1.2 COMPOSITES

1.2.1 Why a Composite?

A composite is a structural material that consists of two or more combined constituents that are combined at a macroscopic level and are not soluble in each other. For many decades composite materials are replacing the other manufacturing materials because of their low cost, easy availability and good mechanical properties. Composites have already proven their worth as weight-saving materials; now the current challenge is to make them cost effective. There have been derived many manufacturing techniques to economically fabricate composite material by the composite industries. Improvement in composite manufacturing technique alone is not sufficient to reduce the cost of the composite significantly hence it is important to pay attention in other phases also like design, material to be used, process of fabrication, tools and machines used, quality control, time of fabrication and also the program management to reduce the overall cost of the composite effectively.

Composite materials have been used widely by the aerospace industries during past several years and now it is shifting rapidly towards commercial use as a manufacturing material for various applications.

Unlike the conventional materials like steel, copper, aluminium etc. the properties of the composite materials can be designed considering structural aspects.

The design of a structural component using composites involves both material and structural design. The variations of composite properties such as stiffness, thermal expansion etc. can be controlled of the designer in a broad range of values. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement.

By the introduction of newer polymer resin matrix materials and high performance reinforcement fibers such as glass, aramid and carbon the use of composite materials as a replacement in complex and advanced materials has gain greater scope. High performance FRP can now be found in such diverse applications

as composite armoring designed resist explosion impacts, windmill blades, fuel cylinders for natural gas vehicles, industrial drive shafts, making rollers and also in bridges For certain applications, the use of composites rather than metals has in fact resulted in savings of both cost and weight. Some examples are curved fairing and fillets, cascades for engines, replacements for welded metallic parts, tubes, cylinders, blade containment bands, ducts etc.

Externally bonded fibre reinforced polymers (FRPs) in the form of continuous glass, carbon, or aramid fibres bonded together in a matrix made of epoxy, polyester or vinylester, are being employed extensively throughout the world as a reinforced concrete structures for retrofitting. They possess properties like high strength-to-weight ratio, highly capable to absorb shocks and vibration, ease of handling and installation, immunity to corrosion etc. and hence FRP jackets are being used increasingly as material of choice for seismic retrofitting projects, even after their relatively high material costs.

Whilst the use of composites will be a clear choice in many cases, material selection in others will depend on factors such as working lifetime requirements, number of items to be produced (run length), complexity of product shape, possible savings in assembly costs and on the experience & skills the designer in tapping the optimum potential of composites. In instances, best results may be achieved through the use of composites in conjunction with traditional materials.

1.2.2 What is a composite?

A typical composite material is a system of materials composing of two or more materials (mixed and bonded) on a macroscopic scale.

Generally, a composite material is composed of reinforcement (fibers, particles, flakes, and/or fillers) embedded in a matrix (polymers, metals, or ceramics). The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed properly, the new combined material exhibits better strength than would each individual material.

As defined by Jartiz, [1] Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different incomposition and characteristics and sometimes in form.

Kelly [2] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength or resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Berghezan [3] defines as "The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their shortcomings", in order to obtain an improved material.

Van Suchetclan [4] explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

1.2.3 Characteristics of the Composites

Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the 'reinforcement' or 'reinforcing material', whereas the continuous phase is termed as the ' matrix'.

Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties.

The shape of the discontinuous phase (which may by spherical, cylindrical, or rectangular cross-sanctioned prisms or platelets), the size and size distribution (which controls the texture of the material) and volume fraction determine the interfacial area, which plays an important role in determining the extent of the interaction between the reinforcement and the matrix.

Concentration, usually measured as volume or weight fraction, determines the contribution of a single constituent to the overall properties of the composites. It is not only the single most important parameter influencing the properties of the composites, but also an easily controllable manufacturing variable used to alter its properties

1.2.4 Classification of Composites

Composite materials can be classified in different ways [5]. Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high performance of the composites. A typical classification is presented in Table-1.1. The two broad classes of composites are:

1. Fibrous composites
2. Particulate composites

1.2.4.1 Particulate Composites

As the name itself indicates, the reinforcement is of particle nature (platelets are also included in this class). It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape, but it is approximately equiaxed. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a limited extent. Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage.

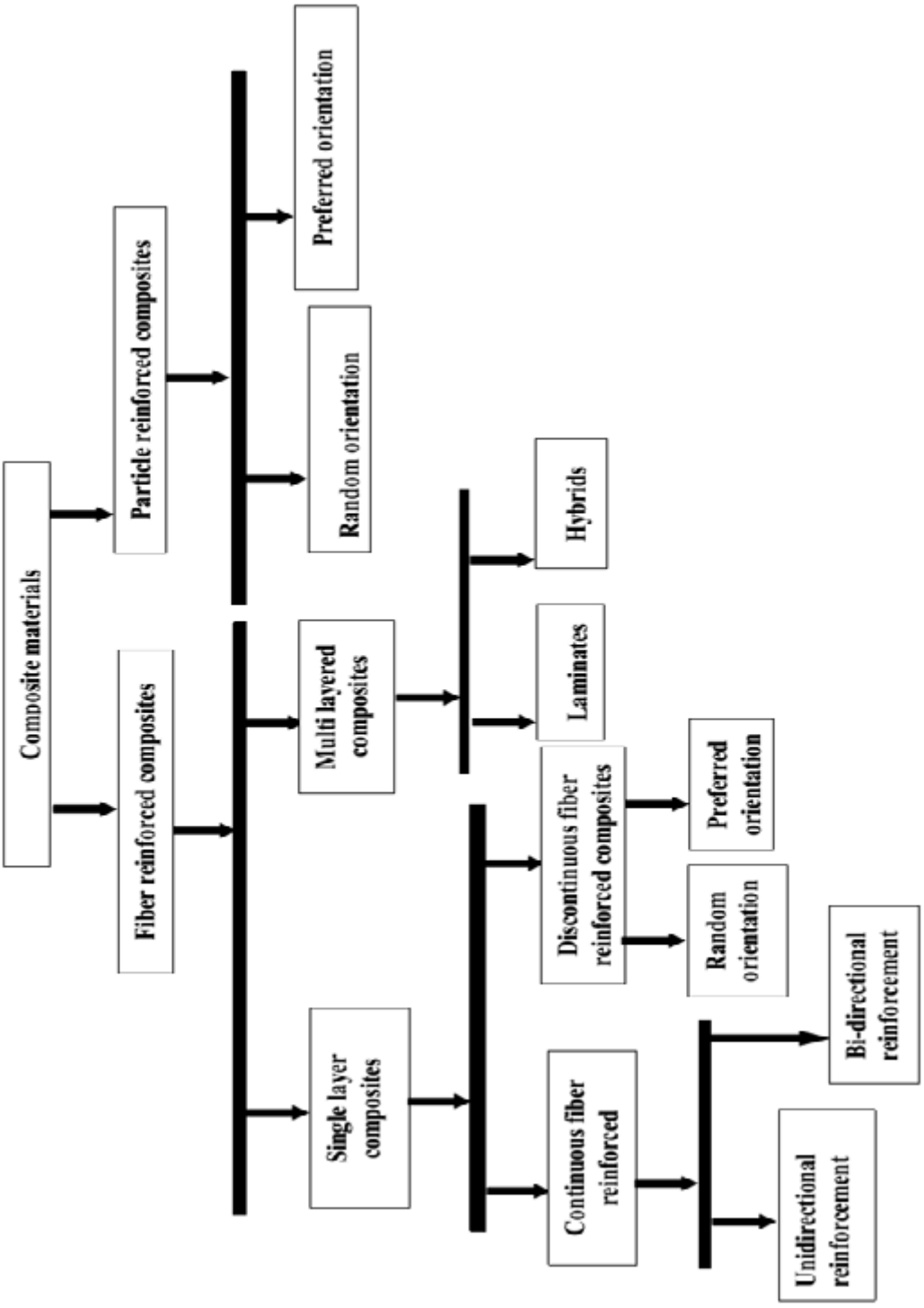
1.2.4.2 Fibrous composites

A fiber is characterized by its length being much greater compared to its cross-sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might otherwise lead to failure, particularly with brittle matrices.

Man-made filaments or fibers of non-polymeric materials exhibit much higher strength along their length since large flaws, which may be present in the bulk

material, are minimized because of the small cross-sectional dimensions of the fiber. In the case of polymeric materials, orientation of the molecular structure is responsible for high strength and stiffness.

Table- 1.1: Classification of composite



Fibers, because of their small cross- sectional dimensions, are not directly usable in engineering applications. They are, therefore, embedded in matrix materials to form fibrous composites. The matrix serves to bind the fibers together, transfer loads to the fibers, and protect them against environmental attack and damage due to handling. In discontinuous fiber reinforced composites, the load transfer function of the matrix is more critical than in continuous fiber composites.

1.3 COMPONENTS OF A COMPOSITE MATERIAL

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the 'matrix'), and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

1.3.1 Role of matrix in a composite

Many materials when they are in a fibrous form exhibit very good strength but to achieve these properties the fibers should be bonded by a suitable matrix. The matrix isolates the fibers from one another in order to prevent abrasion and formation of new surface flaws and acts as a bridge to hold the fibers in place. A good matrix should possess ability to deform easily under applied load, transfer the load onto the fibers and evenly distributive stress concentration.

A study of the nature of bonding forces in laminates [6] indicates that upon initial loading there is a tendency for the adhesive bond between the reinforcement and the matrix to be broken. The frictional forces between them account for the high strength properties of the laminates.

1.3.2 Materials used as matrices in composites

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the matrix) and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

1.3.2.1 Bulk-Phases

a) Metal Matrices

Metal matrix composites possess some attractive properties, when compared with organic matrices. These include (i) strength retention at higher temperatures, (ii) higher transverse strength, (iii) better electrical conductivity, (iv) superior thermal conductivity, (v) higher erosion resistance etc. However, the major disadvantage of metal matrix composites is their higher densities and consequently lower specific mechanical properties compared to polymer matrix composites. Another notable difficulty is the high-energy requirement for fabrication of such composites.

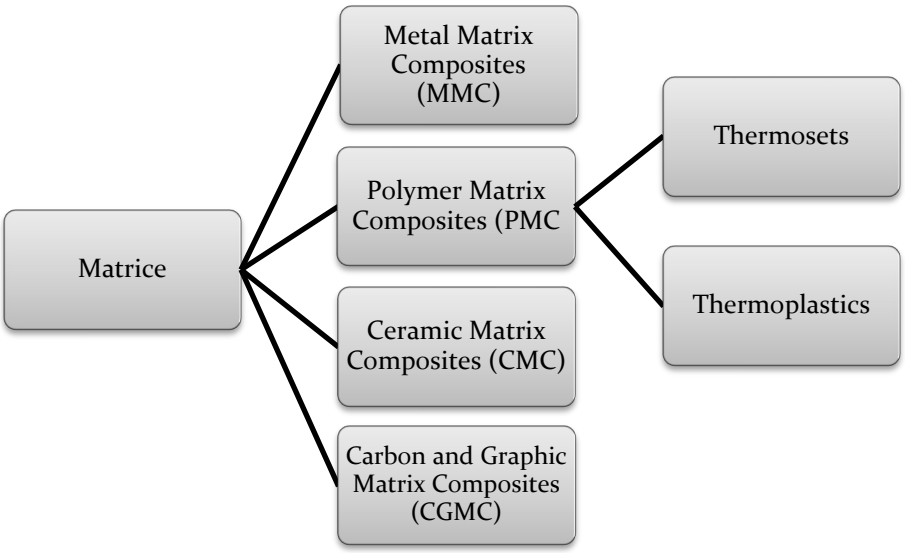


Table- 1.2: Types of marices.

In the aerospace industry interest has been concentrated primarily on fiber reinforced aluminium and titanium. Boron and to a lesser extent silicon carbide (SiC), have been investigated as the reinforcing fibers. Aluminium alloys reinforced with boron have been extensively produced by a variety of methods. Titanium reinforced with SiC, boron (coated with SiC) and beryllium, used for compressor blades.

Good elastic modulus properties can be achieved by the unidirectional incorporation of fibers or whiskers in the metal matrix even though the bonding between them may be poor. But, strong metallic matrices rather than weak metal or polymer matrices are essential for good transverse modulus and shear strength.

Carbon/graphite fibers have been used with metal matrices on a laboratory / experimental scale only, because most basic fabrication techniques involve high

temperatures which have detrimental effects on the fiber. However, research on these lines is continuing in view of the potential of the composites.

b) Polymer Matrices

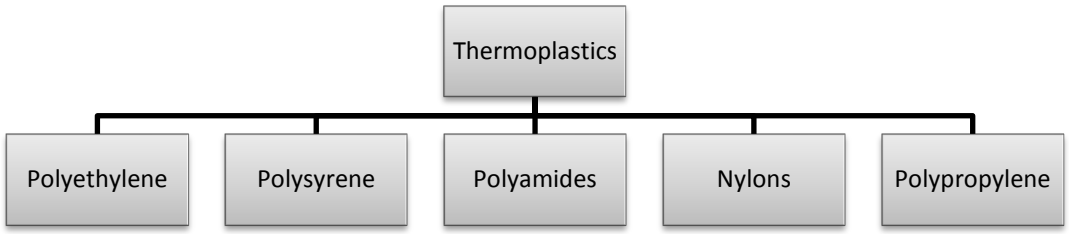
A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the composites. Some of the major advantages and limitations of resin matrices are shown in Table-1.3

Table- 1.3: Advantages and limitations of polymer matrix materials

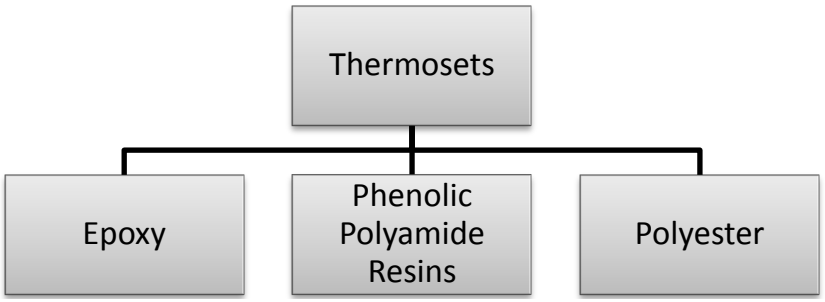
Advantages	Limitations
Low densities	Low transverse strength
Good corrosion resistance	Low operational temperature limits
Low thermal conductivities	
Low electrical conductivities	
Translucence	
Aesthetic Colour effects	

Usually the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance etc. The resin must have mechanical strength commensurate with that of the reinforcement. It must be easy to use in the fabrication process selected and also stand up to the service conditions. Apart from these properties, the resin matrix must be capable of wetting and penetrating into the bundles of fibers which provide the reinforcement, replacing the dead air spaces therein and offering those physical characteristics capable of enhancing the performance of fibers.

Shear, chemical and electrical properties of a composite depend primarily on the resin. Again, it is the nature of the resin that will determine the usefulness of the laminates in the presence of a corroding environment.



(a)



(b)

Table- 1.4: Types of Thermoplastics & Thermosets

Generally speaking, it can be assumed that in composites, even if the volume fraction of the fiber is high (of the order of 0.7), the reinforcement is completely covered by the matrix material; and when the composite is exposed to higher temperatures it is the matrix, which should withstand the hostile environment. Of course, the strength properties of the composite also show deterioration, which may be due to the influence of the temperature on the interfacial bond. Thus, the high temperature resistant properties of the composites are directly related more to the matrix, rather than to the reinforcement. The search for polymers which can withstand high temperatures has pushed the upper limit of the service temperatures to about 300-350°C. This range of operational temperatures can be withstood by polyimides, which are the state-of-the-art high temperature polymers for the present.

Table-1.3 and 1.4 indicate the approximate service temperature ranges for the resins and composites [7, 8]. It should be remembered that there is no place for compromise as to the nature of the matrix material, particularly when it comes to the application temperature of the composite. If the application temperature exceeds 300-350°C metal matrix appears to be the only alternative, at least for the present.

c) Ceramic Matrices

Ceramic fibres, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications, and also where environment attack is an issue. Since ceramics have poor properties in tension and shear, most applications as reinforcement are in the particulate form (e.g. zinc and calcium phosphate). Ceramic Matrix Composites (CMCs) used in very high temperature environments, these materials use a ceramic as the matrix and reinforce it with short fibres, or whiskers such as those made from silicon carbide and boron nitride.

Table- 1.5 Application temperatures of some matrix material

Matrix material	Limit of	
	Long term exposure, °C	Short term exposure, °C
Unsaturated polyesters	70	100
Epoxies	125	200
Phenolics	250	1600
Polyimides	315	400
Aluminium	300	350

Table- 1.6 Trends for temperature application of heat resistant composites

Fiber reinforced Composite	Maximum service temperature, °C	Specific weight gm/cm^3
Carbon / Epoxy	180	1.4
Boron/Epoxy	180	2.1
Borsic / Aluminium	310	2.8
Carbon/Polyimide	310	1.4
Boron/Polyimide	310	2.1
Carbon/Polyaminoxaline	350	1.4
Carbon/Polybenzthiazole	400	1.4
Borsic/Titanium	540	3.6
Carbon/Nickel	930	5.3
Whisker/Metals	1800	2.8-5.6

1.3.2.2 Reinforcement

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. All of the different fibres used in composites have different properties and so affect the properties of the composite in different ways. For most of the applications, the fibres need to be arranged into some form of sheet, known as a fabric, to make handling possible. There are different ways for assembling fibers into sheets and the variety of fiber orientations possible to achieve different characteristics.

1.3.2.3 Interface

It has characteristics that are not depicted by any of the component in isolation. The interface is a bounding surface or zone where a discontinuity occurs, whether physical, mechanical, chemical etc. The matrix material must "wet" the fiber. Coupling agents are frequently used to improve wettability. Well "wetted" fibers increase the interface surfaces area. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the fibers via the interface. This means that the interface must be large and exhibit strong adhesion between fibers and matrix. Failure at the interface (called de-bonding) may or may not be desirable.

1.4 TYPES OF COMPOSITE MATERIALS

The composite materials are broadly classified into the following categories as shown in Figure-1.1

1.4.1 Fiber-reinforced composites

Reinforced-composites are popularly being used in many industrial applications because of their inherent high specific strength and stiffness. Due to their excellent structural performance, the composites are gaining potential also in tribological applications. Fiber reinforced composites materials consists of fiber of high strength and modulus in or bonded to a matrix with distinct interfaces (boundary) between them [4, 5]. In this form both fibers and matrix retain their physical and chemical identities. Yet they produce a combination of properties that cannot be achieved with either of the constituents acting alone. In general, fibers are the principal load carrying candidates, while the surrounding matrix keeps them in the desired location and

orientation [5, 6]. A Fibrous composite can be classified into two broad groups: continuous (long) fiber composite and discontinuous (short) fiber composite.

1.4.1.1 Continuous or long fiber composite

Continuous or long fiber composite consists of a matrix reinforced by a dispersed phase in the form of continuous fibers. A continuous fiber is geometrically characterized as having a very high length-to- diameter ratio. They are generally stronger and stiffer than bulk material. Based on the manner in which fibers are packed within the matrix, it is again subdivided in to two categories: (a) unidirectional reinforcement and (b) bidirectional reinforcement. In unidirectional reinforcement, the fibers are oriented in one direction only where as in bidirectional reinforcement the fibers are oriented in two directions either at right angle to one another (cross-ply), or at some desired angle (angle-ply). When fibers are large and continuous, they impart certain degree of anisotropy to the properties of the composites particularly when they are oriented. Multi-axially oriented continuous fiber composites are also display near isotropic properties.

1.4.1.2 Discontinuous or short fiber composite

Short-fiber reinforced composites consist of a matrix reinforced by a dispersed phase in form of discontinuous fibers (length < 100×diameter). The low cost, ease of fabricating complex parts, and isotropic nature are enough to make the short fiber composites the material of choice for large-scale production. Consequently, the short-fiber reinforced composites have successfully established its place in lightly loaded component manufacturing. Further the discontinuous fiber reinforced composite divided into: (a) biased or preferred oriented fiber composite and (b) random oriented fiber composite. In the former, the fibers are oriented in predetermined directions, whereas in the latter type, fibers remain randomly. The orientation of short fibers can be done by sprinkling of fiber on to given plane or addition of matrix in liquid or solid state before or after the fiber deposition. The discontinuities can produce a material response that is anisotropic, but the random reinforcement produces nearly isotropic properties.

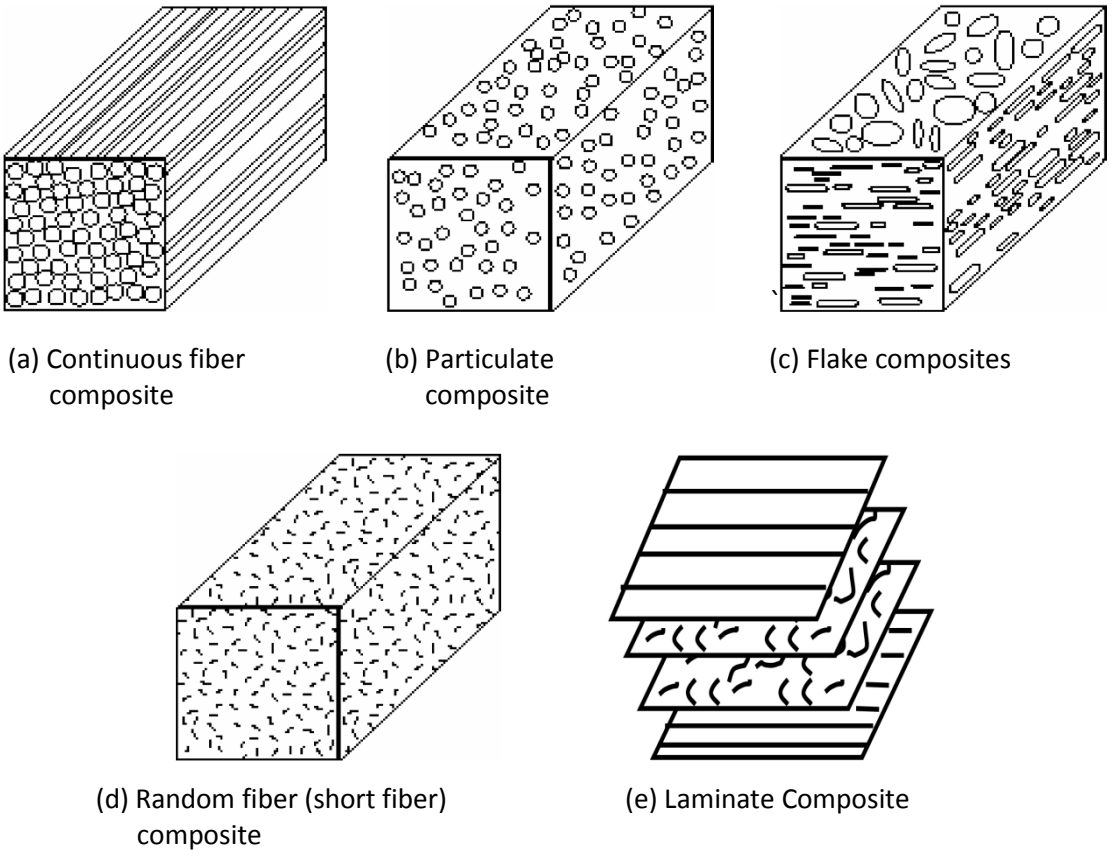


Figure-1.1 Schematic diagram of different types of Composite

1.4.2 Laminate Composites

Laminate Composites are composed of layers of materials held together by matrix. Generally, these layers are arranged alternatively for the better bonding between reinforcement and the matrix. These laminates can have unidirectional or bi-directional orientation of the fiber reinforcement according to the end use of the composite. Composite laminates are of different types such as unidirectional, angle-ply, cross-ply and symmetric laminates. A hybrid laminate can also be fabricated by the use of different constituent materials or of the same material with different reinforcing pattern. In most of the applications of laminate composites, man-made fibers are used due to their good combination of physico-mechanical and thermal behavior.

1.5 THESIS OUTLINE

The remainder of this thesis is organized as follows:

Chapter 2: Previous work relevant to the present investigations available in literatures is described.

Chapter 3: This chapter describes the details of materials required, fabrication techniques and the results from the tests for mechanical properties and erosion wear behavior of the developed composite.

Chapter 4: In this chapter the fiber surface modification by chemical methods namely, acetone and alkali has been carried out. The improvement in the mechanical and erosion wear behavior after fiber treatment has been reported.

Chapter 5: Conclusions from the above work has been drawn and recommendations and scope for future work are presented in this chapter.

Chapter-2

2.1 LITERATURE SURVEY

Literature survey is carried out to get the background information on the issues to be considered in the present research work and to focus the relevance of the present study. The purpose is also to present a thorough understanding of various aspects of natural fiber polymer composite with a special attention to their mechanical properties and erosion wear behavior.

2.2 NATURAL FIBERS: Initiative in Product Development.

In fiber reinforced polymer composites, the fibers can be either synthetic fibers or natural fibers. Natural fibers constituents are mainly of cellulose fibers, consisting of helically wound cellulose micro fibrils, bound together by an amorphous lignin matrix. Lignin keeps the water in fibers; acts as a protection against biological attack and as a stiffener to give stem its resistance against gravity forces and wind. Hemicellulose found in the natural fibers is believed to be a compatibilizer between cellulose and lignin [9].The use of lignocellulosic fibers as reinforcements for polymeric materials has been growing during the last decade or so to replace synthetic fibers, especially glass fibers in composites, for different industrial sectors, such as packaging, automobiles [10, 11] and even in the building sector [12]. This is mainly due to their unique characteristics, such as availability, biodegradability, low density, non-toxic nature, less abrasiveness to plastic processing equipment, useful mechanical properties and low cost [13]. The physical mechanical properties of natural fibers are greatly influenced by their chemical compositions. The properties of some of these fibers are presented in Table-2.1 [14]. It is evident from Table-2.1 that, the tensile strength of glass fiber is substantially higher than that of natural fibers even though the modulus is of the same order. However, when the specific modulus of natural fibers is considered, the natural fibers are better as compared to glass fibers. Therefore, these higher specific properties are the major advantages of natural fiber as reinforcement in polymer composites for weight sensitive applications.

Table 2.1 Properties of natural fibers [14]

Fiber	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Density (g/cm^3)
Abaca	400	12	3-10	1.5
Alfa	350	22	5.8	0.89
Bagasse	290	17	-	1.25
Bamboo	140-230	11-17	-	0.6-1.1
Banana	500	12	5.9	1.35
Coir	175	4-6	30	1.2
Cotton	287-597	5.5-12.6	7-8	1.5-1.6
Curaua	500-1,150	11.8	3.7-4.3	1.4
Palm	97-196	2.5-5.4	2-4.5	1-1.2
Flax	345-1,035	27.6	2.7-3.2	1.5
Hemp	690	70	1.6	1.48
Henequen	500±70	13.2 ± 3.1	4.8 ± 1.1	1.2
Isora	500-600	-	5-6	1.2-1.3
Jute	393-773	26.5	1.5-1.6	1.3
Kenaf	930	53	1.6	-
Nettle	650	38	1.7	-
Oil palm	248	3.2	25	0.7-1.55
Piassava	134-143	1.07-4.59	21.9-7.8	1.4
Pineapple	400-627	1.44	14.5	0.8-1.6
Ramie	560	24.5	2.5	1.5
Sisal	511-635	9.4-22	2.0-2.5	1.5
E-Glass	3400	72	-	2.5

Natural organic fibers can be derived from either animal or plant sources. The majority of useful natural textile fibers are plant derived, with the exceptions of wool and silk. All plant fibers are composed of cellulose, whereas fibers of animal origin consist of proteins. Natural fibers in general can be classified based on their origin, and the plant-based fibers can be further categorized based on part of the plant they are recovered from. An overview of natural fibers is presented in Figure-2.1 [15].

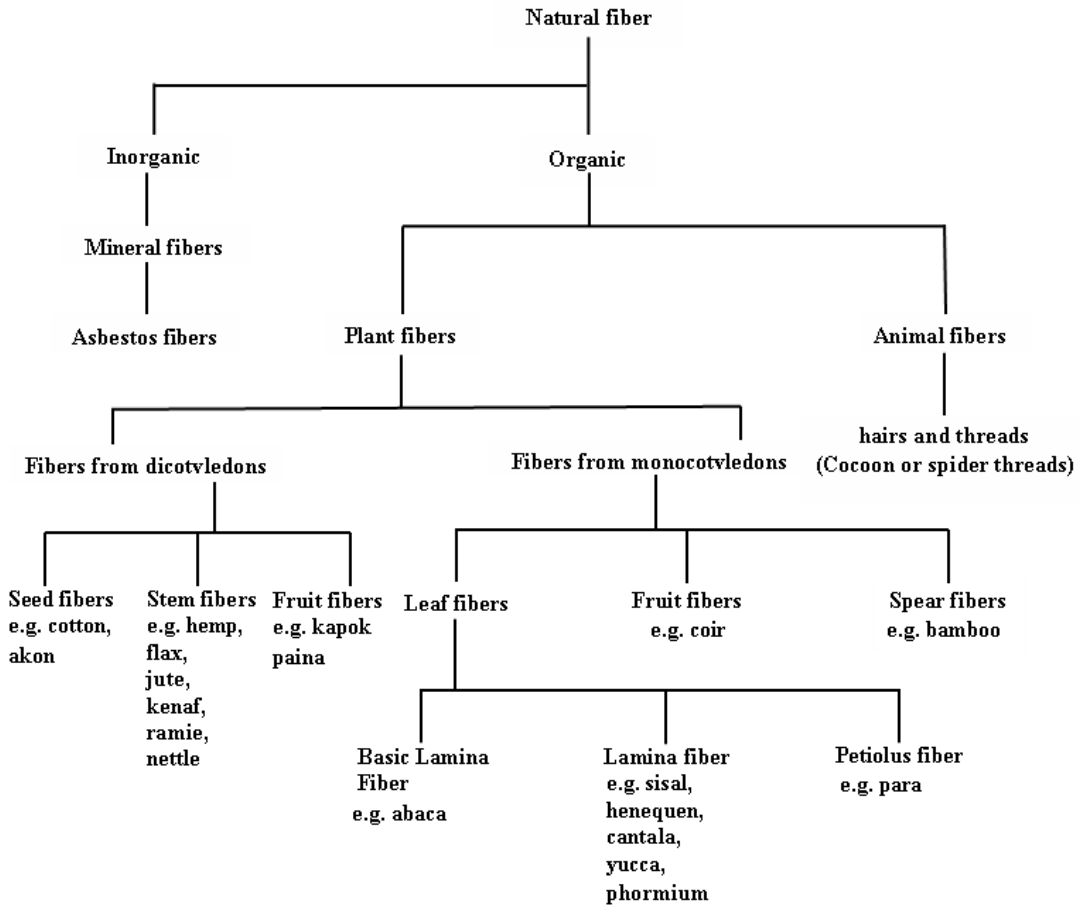


Figure-2.1: Overview of natural fiber [15].

A great deal of work has already been done on the effect of various factors on mechanical behavior of natural fiber reinforced polymer composites. The post-impact behavior of jute fiber reinforced polyester composites subjected to low velocity impact has been studied by Santulli [16]. Effect of fiber content on tensile and flexural properties of pineapple fiber reinforced poly (hydroxybutyrate-co-valerate) resin composites has studied by Luo and Netravali [17].The mechanical behavior of jute and kenaf fiber reinforced polypropylene composites has been studied by Schneider and Karmaker [18]. It is concluded from their study that jute fiber based composites provides better mechanical properties than kenaf fiber based composites. The effect of various loading rate on mechanical properties of jute/glass reinforced epoxy based hybrid composites has studied by Srivastav et al. [19]. The mechanical properties of jute fiber reinforced polyester composites were evaluated by Gowda et al. [20]. It is reported from their study that jute fiber based composites have better strengths as compared to wood based composites. The limited use of natural fiber composites is also connected with some other major disadvantages still associated

with these materials. Furthermore, the fibers inherent hydrophilic nature makes them susceptible to water uptake in moist conditions. Natural fiber composites tend to swell considerably with water uptake and as a consequence mechanical properties, such as stiffness and strength, are negatively influenced. However, the natural fiber is not inert. The fiber-matrix adhesion may be improved and the fiber swelling reduced by means of chemical, enzymatic or mechanical modifications [21].

S.K.Acharya et al. [22] studied the influence of fiber treatment on the performance of bagasse fiber reinforced polymer composite by different chemical treatments. They reported considerable improvement on the mechanical properties of the composite. Their report states that the shear stress of the composite is very sensitive to the treatments. The shear stress decreases with increasing in fiber weight fraction.

During the last few years, a series of works have also been done to replace the conventional synthetic fiber with natural fiber composites [23, 24, 25-30]. Nevertheless, certain aspects of natural fiber reinforced composite behavior is still poorly understood such as their visco elastic, visco plastic or time-dependent behavior due to creep and fatigue loadings [31], interfacial adhesion [32, 33], and tribological properties. Hashmi et al.[34] investigated the sliding wear behavior of cotton–polyester composites and obtained better wear properties on addition of cotton reinforcement. Tong et al. [35] studied the abrasive wear behavior of bamboo and reported that the abrasive resistance of a bamboo stem is affected by the vascular bundle fiber orientation with respect to the abrading surface and the abrasive particle size. Recently, Chand and Dwivedi [36] report that the maleic-anhydride-grafted polypropylene improved the wear properties of jute–polypropylene composites. In another paper, they studied the tribological behavior of Wood Flour loading on epoxy composites [37], and found that Wood Flour loading increases the load carrying capacity of epoxy and decrease its wear resistance. Tayeb [38] reports the tribo-potential of sugarcane fiber reinforcement in the thermoset polymers for enhancing the adhesive wear resistance. Likewise rice husk has also considerable potential as reinforcement for polymer and may provide advantage when used as a substitute for conventional synthetic fiber. Recently Navin chand et al [39] studied the positive effect of rice husk reinforcement as well as surface treatment on tribological and mechanical properties of the resulting PVC-based composite.

In another work vinay kumar et al [40] reports that Polymer composites based on rice husk and polypropylene can be made by compression or an injection molding process. The amount of rice husk that can be utilized up to 40–60% resulting in significant reduction in cost but higher amounts can cause handling and agglomeration problems**.**

The conclusions drawn from this is that, the success of combining rice husk with polymer matrices results in the improvement of mechanical properties of the composites compared with the matrix materials. These fillers are cheap and nontoxic, can be obtained from renewable sources, and are easily recyclable. Moreover, despite their low strength, they can lead to composites with high specific strengths because of their low density.

2.3 OBJECTIVE OF THE RESEARCH WORK

The priority of this work is to prepare polymer Matrix Composites (PMCs) using rice husk (waste from rice mill industry) as reinforcement material. In the present work two series of composites were prepared using randomly oriented unmodified and modified rice husk as reinforcement in epoxy matrix. The composite are prepared with different concentration of rice husk and the erosive wear behavior and mechanical properties of the composites has been studied with different fiber loading.

Figure-2.2: Photograph of Rice Husk

Chapter-3

3.1 INTRODUCTION

Wear is a characteristic of the engineering system which depends on load, speed, temperature, hardness, presence of foreign material and the environmental condition [41]. Widely varied wearing conditions cause wear of materials. It may be due to surface damage or removal of material from one or both of two solid surfaces in a sliding, rolling or impact motion relative to one another. In most cases wear occurs through surface interactions at asperities. During relative motion, material on contacting surface may be removed from a surface, may result in the transfer to the mating surface, or may break loose as a wear particle. The wear resistance of materials is related to its microstructure may take place during the wear process and hence, it seems that in wear research emphasis is placed on microstructure [42]. Wear of material depends on many variables, so wear research program must be planned systematically. Therefore researchers have normalized some of the data to make them more useful. The wear map proposed by Lim [43] is very much useful in this regard to understand the wear mechanism in different sliding conditions as well as the anticipated rates of wear.

3.2 THEORY OF WEAR

Wear occurs as a natural consequence when two surfaces with a relative motion interact with each other. Wear may be defined as the progressive loss of material from contacting surfaces in relative motion. Scientists have developed various wear theories in which the Physico-Mechanical characteristics of the materials and the physical conditions (e.g. the resistance of the rubbing body and the stress state at the contact area) are taken in to consideration. In 1940 Holm [44] starting from the atomic mechanism of wear, calculated the volume of substance worn over unit sliding path.

Barwell and Strang [45] in 1952: Archard [46] in 1953 and Archard and Hirst [47] in 1956 developed the adhesion theory of wear and proposed a theoretical equation identical in structure with Holm's equation. In 1957, Kragelski [48] developed the fatigue theory of wear. This theory of wear has been widely accepted by scientists in different countries. Because of the Asperities in real bodies, their interactions in

sliding is discrete, and contact occurs at individual locations, which, taken together, form the real contact area. Under normal force the asperities penetrate into each other or are flattened out and in the region of real contact points corresponding stress and strain rise. In sliding, a fixed volume of material is subjected to the many times repeated action, which weakens the material and leads finally to rupture. In 1973, Fleischer [49] formulated his energy theory of wear. The main concept of this theory is that the separation of wear particles requires that a certain volume of material accumulates a specific critical store of internal energy. It is known that a large part of the work done in sliding is dissipated as heat, and that a small proportion of it accumulates in the material as internal potential energy. When the energy attains a critical value, plastic flow of the material occurs in this volume or a crack is formed. Further theories of wear are found in [48]. Though all the theories are based on different mechanisms of wear, the basic consideration is the frictional work.

With regards to the usage of natural fiber as reinforcement for tribological application in polymeric composite, few works have been attempted. However, in recent years, some work has been done on natural fiber like jute [50], cotton [51, 52], oil palm [53], coir [54], kenaf [55], betel-nut [56], betel palm [57], wood flour [58] and bamboo powder [59] as reinforcement. In these works, the wear resistance of polymeric composites has been improved when natural fiber introduced as reinforcement.

3.3 TYPES OF WEAR

Wear may be classified as:-

(1) Abrasive (2) Adhesive (3) Erosive (3) Surface fatigue (4) Corrosive

3.3.1 Abrasive wear

Abrasive wear may be defined as the wear that occurs when a hard surface slides against and cuts groove from a softer surface. It can be account for most failures in practice. Hard particles or asperities that cut or groove one of the rubbing surfaces produce abrasive wear. This hard material may be originated from one of the two rubbing surfaces. In sliding mechanisms, abrasion can arise from the existing asperities on one surface (if it is harder than the other), from the generation of wear fragments which are repeatedly deformed and hence get work hardened for oxidized until they became harder than either or both of the sliding surfaces, or from the

adventitious entry of hard particles, such as dirt from outside the system. Two body abrasive wear occurs when one surface (usually harder than the second) cuts material away from the second, although this mechanism very often changes to three body abrasion as the wear debris then acts as an abrasive between the two surfaces. Abrasives can act as in grinding where the abrasive is fixed relative to one surface or as in lapping where the abrasive tumbles producing a series of indentations as opposed to a scratch. According to the recent tribological survey, abrasive wear is responsible for the largest amount of material loss in industrial practice [60].

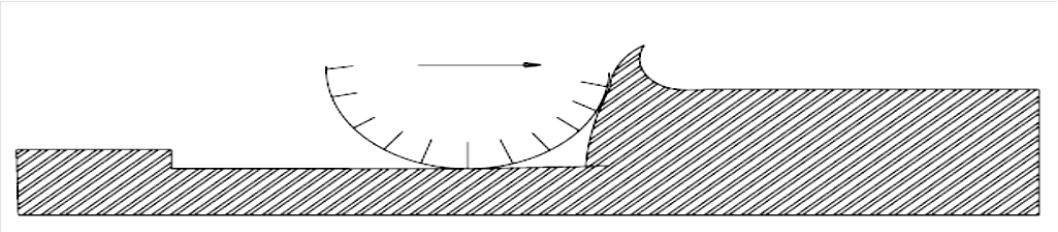


Figure-3.1 : Schematic representations of the abrasion wear mechanism

3.3.2 Adhesive wear

Adhesive wear can be defined as the wear due to localized bonding between contacting solid surfaces leading to material transfer between the two surfaces or the loss from either surface. For adhesive wear to occur it is necessary for the surfaces to be in intimate contact with each other. Surfaces, which are held apart by lubricating films, oxide films etc. reduce the tendency for adhesion to occur.

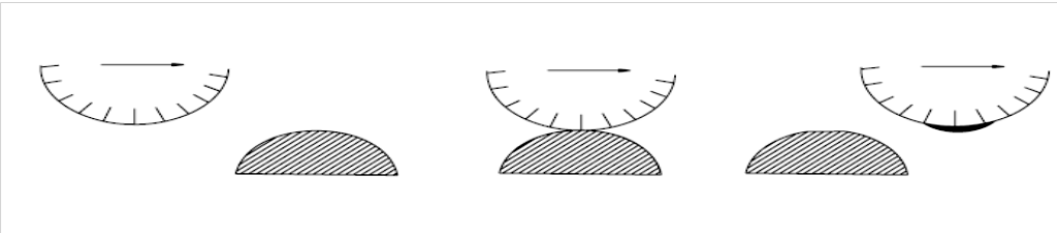


Figure-3.2: Schematic representations of the adhesive wear mechanism

3.3.3 Erosive wear

Erosive wear can be defined as the process of metal removal due to impingement of solid particles on a surface. Erosion is caused by a gas or a liquid, which may or may not carry, entrained solid particles, impinging on a surface. When the angle of impingement is small, the wear produced is closely analogous to abrasion. When the

angle of impingement is normal to the surface, material is displaced by plastic flow or is dislodged by brittle failure.

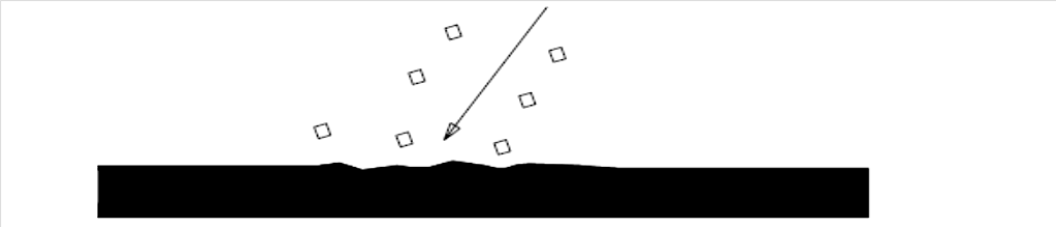


Figure-3.3: Schematic representations of the erosive wear mechanism

3.3.4 Surface fatigue wear

Wear of a solid surface is caused by fracture arising from material fatigue. The term 'fatigue' is broadly applied to the failure phenomenon where a solid is subjected to cyclic loading involving tension and compression above a certain critical stress. Repeated loading causes the generation of micro cracks, usually below the surface, at the site of a pre-existing point of weakness. On subsequent loading and unloading, the micro crack propagates. Once the crack reaches the critical size, it changes its direction to emerge at the surface, and thus flat sheet like particles is detached during wearing. The number of stress cycles required to cause such failure decreases as the corresponding magnitude of stress increases. Vibration is a common cause of fatigue wear.

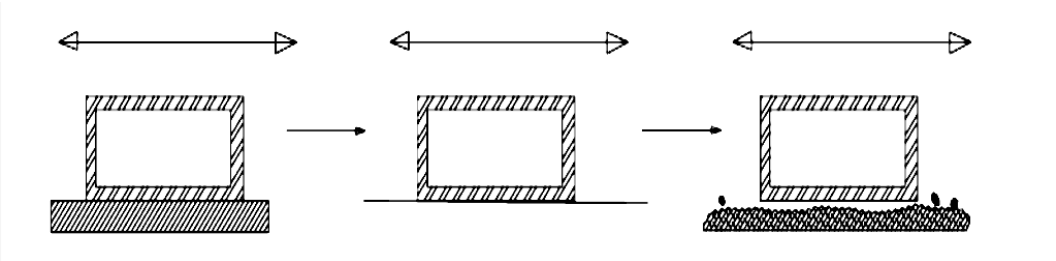


Figure-3.4: Schematic representations of the surface fatigue wear mechanism

3.3.5 Corrosive wear

Most metals are thermodynamically unstable in air and react with oxygen to form an oxide, which usually develop layer or scales on the surface of metal or alloys when their interfacial bonds are poor. Corrosion wear is the gradual eating away or deterioration of unprotected metal surfaces by the effects of the atmosphere, acids,

gases, alkalis, etc. This type of wear creates pits and perforations and may eventually dissolve metal parts.

3.4 SYMPTOMS OF WEAR

A summary of the appearance and symptoms of different wear mechanism is indicated in Table–3.1 and the same is a systematic approach to diagnose the wear mechanisms.

Table-3.1 Symptoms and appearance of different types of wear [61].

Types of wear	Symptoms	Appearance of the worn out surface
Abrasive	Presence of clean furrows cut out by abrasive particles.	Grooves
Adhesive	Metal transfer is the prime symptoms.	Seizure, catering rough and torn-out surfaces.
Erosion	Presence of abrasives in the fast moving fluid and short abrasion furrows.	Waves and troughs.
Corrosion	Presence of metal corrosion products.	Rough pits or depressions.
Fatigue	Presence of surface or subsurface cracks accompanied by pits and spalls.	Sharp and angular edges around pits.
Impacts	Surface fatigue, small sub-micron particles or formation of spalls.	Fragmentation, peeling and pitting.
Delamination	Presence of subsurface cracks parallel to the surface with semi-dislodged or loose flakes.	Loose, long and thin sheet like particles
Fretting	Production of voluminous amount of loose debris.	Roughening, seizure and development of oxide ridges
Electric attack	Presence of micro craters or a track with evidence of smooth molten metal.	Smooth holes

Literature available on the rate of controlling abrasive wear mechanism demonstrates that it may change abruptly from one another at certain sliding velocities and contact loads, resulting in abrupt increases in wear rates. The conflicting results in the abrasive wear literature arise partly because of the differences in testing conditions, but they also make clear that a deeper understanding of the abrasive wear mechanism is required if an improvement in the wear resistances of the polymer matrix composites is to be achieved. This in turn requires a systematic study of the wear under different loads and velocities. It is generally recognized that abrasive wear is a characteristic of a system and is influenced by many parameters. Laboratory scale investigation if designed properly allows careful control of the tribo system whereby the effects of different variables on wear behavior of PMCs can be isolated and determined. The data generated through such investigation under controlled conditions may help in correct interpretation of the results.

As new developments are still under way to explore innovative fields for tribo-application of natural fiber base materials, in this chapter an attempt has been made to study the potential of using rice husk fiber for tribological and mechanical applications. In the current study the effect of fiber loading, sliding velocity, sliding distance and normal load on abrasive wear behavior of rice husk fiber filled epoxy composite has been evaluated and possible wear mechanism has been discussed with SEM observation.

3.5 FABRICATION OF COMPOSITES

For preparation of composites to conduct mechanical and wear test the following materials has been used;

- Rice husk fibers
- Epoxy
- Hardner

3.5.1 Rice husk

Rice husk (RH) is an agricultural waste material abundantly available in rice-producing countries. They are the natural sheaths that form on rice grains during their growth and removed during the refining of rice, these husks have no commercial interest. Rice husk is a fibrous material and has a varied range of aspect ratio. Thus it can be used as filler for making light weight polymer composites. Keeping this in

view the present work has been under taken to develop a polymer matrix composite (epoxy resin).

3.5.2 Epoxy Resin & Hardner

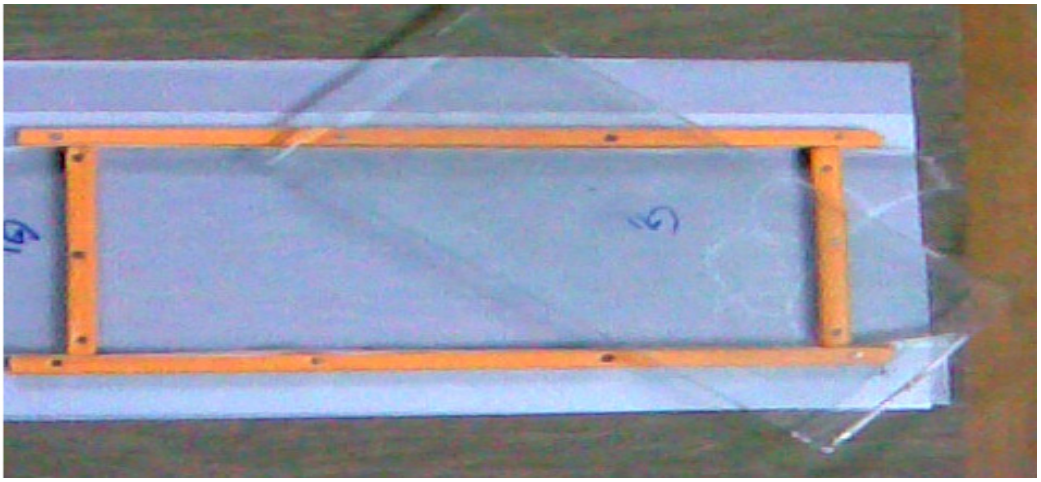
The type of epoxy resin used in the present investigation is Araldite LY-556 which chemically belongs to epoxide family. Its common name is Bisphinol-A-Diglycidyl-Ether. The hardener with IUPAC name NNO-bis (2aminoethylethane-1,2diamin) has been used with the epoxy designated as HY 951. Both the epoxy and hardener were supplied by Ciba Geigy of India Ltd.

3.6 FABRICATION & PREPARATION OF COMPOSITE SLABS

The Rice husks were collected locally. They were washed several times with plain water to remove the dust and other foreign particles adherence to the fibers and were dried in sun light. Then these rice husk fibers sieved with sieve shaker and a particular size (i.e. 2.36mm) rice husk fiber is chosen for the experiment.

Usual hand lay-up technique was used for preparation of the samples. A plastic mold of dimension (140x100x6) mm was used for casting the composite sheet (Fig.3.5). A mold release spray was applied at the inner surface of the mold for quick and easy release of the composite sheet. For different weight fraction of fibers, a calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed in a glass jar and placed in a vacuum chamber to remove air bubbles that got introduced. Then calculated amount of rice husk is added to the mixture of epoxy resin and hardner and mixed properly. Then the composite mixture is poured in to the mold.

Care has been taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 72 hrs. During application of pressure some amount of epoxy and hardener squeezes out. Care has been taken to consider this loss during manufacturing so that a constant thickness of sample can be maintained. This procedure was adopted for preparation of 5, 10, 15 and 20% weight fractions of fiber reinforced epoxy composite slabs. After 72 hrs the samples were taken out from the mold and then cut in to required sizes as per ASTM standards for Mechanical test.



(a)

(b)

(c)

Figure-3.5: (a) Mold used for composite preparation (b) Specimen cut for bend test (c) Specimen cut for erosion test.

3.7 TENSILE TEST

The standard test method according to ASTM D3039-76 has been used; gauge length of the test specimen used is 50 mm. The tensile test has been performed in universal testing machine INSTRON H10KS .The test was conducted with a cross head speed of 5mm/min. For each test, composite of five samples were tested and average value was taken for analysis. Figure 3.6 shows the Machine used for the test and the sample in loading condition. The results obtained from the tests are presented in Table-3.2. Tensile strength & Young's modulus were found out using the following formula.

$$\sigma = \frac{P}{A} \quad \text{..... (3.1)}$$

$$E = \frac{\sigma L}{\delta} \quad \text{..... (3.2)}$$

Where ‘σ’ is tensile strength, ‘P’ is the load applied, ‘A’ is the cross sectional area, ‘E’ is Young’s modulus, ‘L’ is span length of the specimen and ‘δ’ is the deflection recorded.

Figure-3.6: INSTRON H10KS TESTING MACHINE

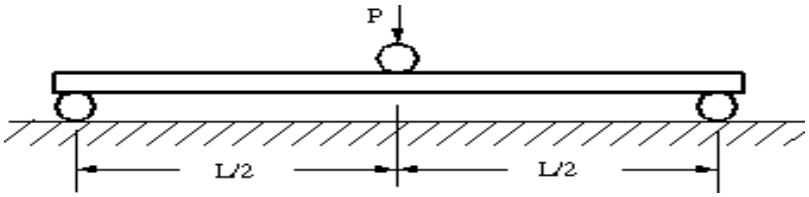
3.8 BEND TEST

Three point bend test was carried out in an UTM 201 machine in accordance with ASTM D2344-84 to measure the flexural strength of the composites. The loading arrangement for the specimen and the photograph of the machine used are shown in Figure-3.7(a) and (b) respectively. All the specimens (composites) were of rectangular shape having length varied from 100-125 mm, breadth of 20-25 mm and thickness of 3.5-5.0 mm. A span of 100 mm was employed maintaining a cross head speed of 5mm/min. The flexural strength in a three point bending test is found out by using equation (3.3). The flexural strength of composites was found out using the following equation

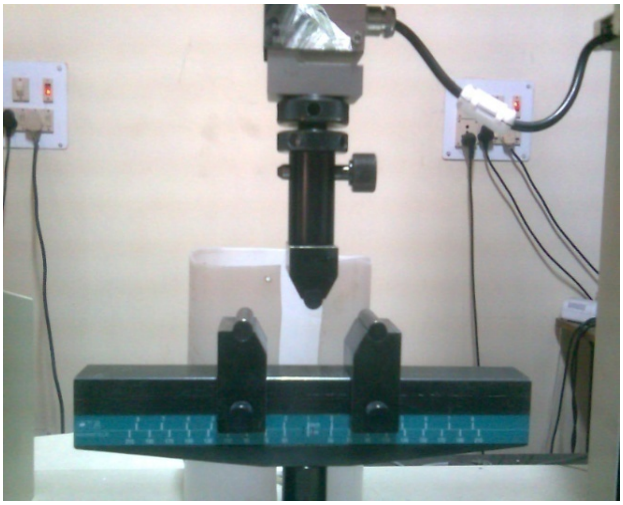
$$FS = \frac{3PL}{2bt^2} \quad \text{..... (3.3)}$$

$$G = \frac{3PL^3}{4bt^3y} \quad \text{..... (3.4)}$$

Where ‘b’ is breadth, ‘t’ is thickness and ‘y’ is extension of the specimen during experiment.



(a)



(b)

Figure-3.7: Loading position of specimen for bend test

3.9 RESULTS OF TENSILE TEST AND BEND TEST

The tensile strength results for various specimens were plotted in figure 3.8.Plot shows that, with increase of fiber concentration the tensile strength goes on increasing and samples with 15% fiber weight fraction gives maximum strength.

Figure 3.9 shows the variation in flexural strength for neat epoxy and different weight fraction of fiber composites. The flexural strength results for different weight

fraction of fiber composite has also follow the same trend as for the tensile strength and have a maximum value for 15% weight fraction of fiber.

Table-3.2 Mechanical properties of PRH reinforced epoxy & Neat Epoxy composites.

Fiber content by weight	Tensile Strength (MPa)	Flexural Strength (MPa)
0%	14.07	18.18
5%	15.37	19.55
10%	17.81	20.29
15%	18.52	22.88
20%	15.55	18.75

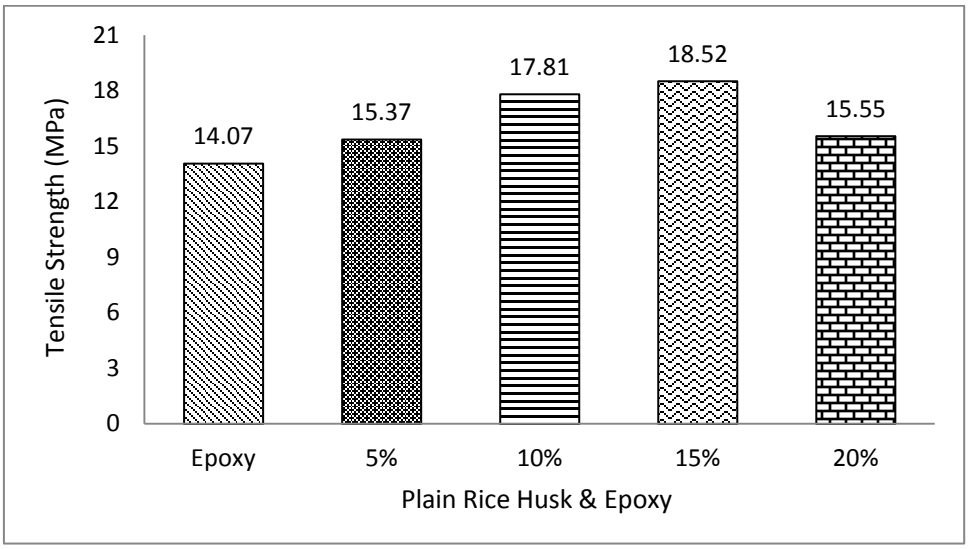


Figure-3.8: Variation of tensile strength with increasing weight % of rice husk reinforcement in the composite.

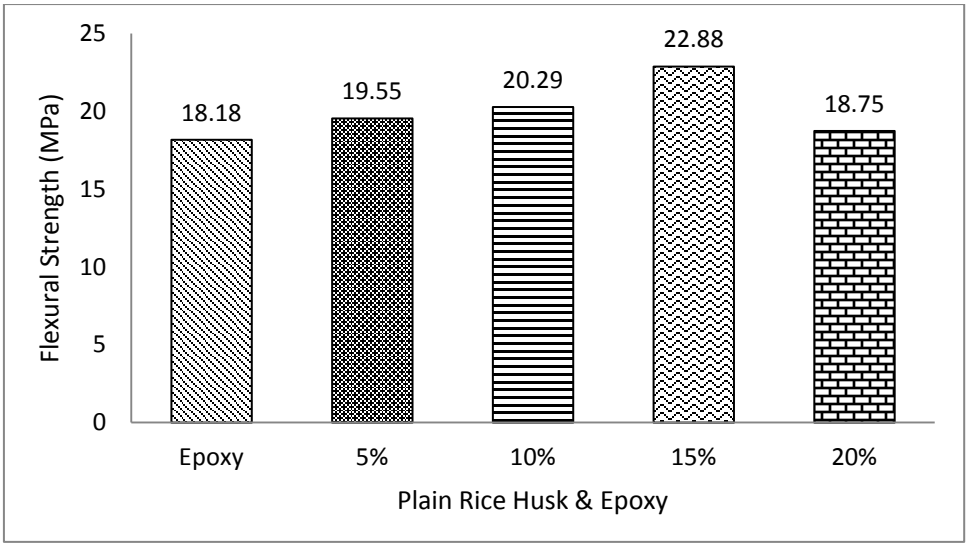


Figure-3.9: Variation of flexural strength with increasing weight % of rice husk reinforcement in the composite.

3.10 SOLID PARTICLE EROSION TEST

Solid particle erosion manifests itself in thinning of components, surface roughening, surface degradation, macroscopic scooping appearance and reduction in functional life of the structure. Hence, solid particle erosion has been considered as a serious problem as it is responsible for many failures in engineering applications. Several attempts to understand the basic mechanisms of the erosion were started in the last half of the 20th century and have been continued to the present. In the year of 1995 an article on the past and the future of erosion was presented by Finnie [62]. In this article, the influencing parameters and dominating mechanisms during solid particle erosion were reviewed on the erosion response of metals and ceramic materials. In the same year another article was published by Meng et al. [63] to provide information about the existing wear models and prediction equations.

The subject of erosion wear of polymer composite has received substantial attention in the past decades. Interest in this area is commensurate with the increasing utilization of polymer based composites in aerospace, transportation and processing industries, where they can be subjected to multiple solid or liquid particle impact. Examples of such applications are pipe lines carrying sand slurries in petroleum refining, helicopter rotor blades , pump impeller blades, high speed vehicles and aircraft operating in desert environments, radomes, surfing boats where the

component encounter impact of lot of abrasives like dust, sand, splinters of materials, slurry of solid particle and consequently the materials undergo erosive wear [64-66].

Many researchers have evaluated the resistance of various types of polymers like nylon, epoxy, polypropylene, bismileimide, etc and their composites to solid particle erosion. Harsha et al. [67] has summarized the work done by some of the investigators on solid particle erosion of polymer composites. Roy et al. while working on erosive wear of polymer composite revealed that the composite materials present a rather poor erosion resistance as compared to metallic materials [68]. Biswas et al. [69] worked on red mud filled glass epoxy composite and explained the ductile and brittle behavior criteria of the composite based on the impact angle of erosion wear.

The most important factors influencing the erosion rate of the composite materials can be summarized under four categories; (i) The properties of the target materials (matrix material properties and morphology, reinforcement type, amount and orientation, interface properties between the matrices and reinforcements, etc.), (ii) Environment and testing conditions (temperature, chemical interaction of erodent with the target), (iii) Operating parameters (angle of impingement, impinging velocity, particle flux–mass per unit time, etc.) and (iv) The properties of the erodent (size, shape, type, hardness, etc.) [66, 70-72]. The ductile behavior is characterized by maximum erosion at low impact angle in the range of 15°–30°. On the other hand, if maximum erosion occurs at 90°, then the behavior can be termed as brittle. Reinforced composites have also been some time found to exhibit an intermediate behavior known as semi-ductile with maximum erosion occurring at an angle in the range of 45°–60° [73]. However, the above classification is not absolute as the erosion behavior of a material has a strong dependence on erosion conditions such as impact angle, impact velocity and erodent properties such as shape, hardness, size etc. In the literature, the erosion behavior of polymers and its composites has also been characterized by the value of the velocity exponent, '*n*' ($E_r \alpha v^n$) [64].

3.11 EXPERIMENT

3.11.1 Preparation for the test specimens

The preparation of the test specimens were carried out as per the procedure discussed in Art-3.5. Specimens of dimension 30 x 30 x 3.0 mm were cut from the

composite slabs. Adequate care has been taken to keep the thickness constant (3mm) for all the samples.

3.11.2 Test apparatus & Experiment

The schematic figure of the erosion test apparatus used for the present investigation designed as per ASTM-G76 standard is shown in Figure-3.10. The rig consists of an air compressor, a particle feeder, and an air particle mixing and accelerating chamber. The compressed dry air is mixed with the erodent particles, which are fed at a constant rate from a conveyor belt-type feeder in to the mixing chamber and then accelerated by passing the mixture through a tungsten carbide converging nozzle of 4 mm diameter. These accelerated particles impact the specimen, and the specimen could be held at various angles with respect to the impacting particles using an adjustable sample holder. The test apparatus has also been fitted with a rotating double disc to measure the velocity of the erodent particle. The impact velocities of the erodent particles has been evaluated experimentally using this rotating double disc method developed as explained by Ives and Ruff [74]. The velocities obtained from this method for various pressures are given in Table-3.3.

The conditions under which the erosion test has been carried out are given in Table-3.4. A standard test procedure is employed for each erosion test. The samples are cleaned in acetone, dried and weighed to an accuracy of 1×10^{-3} gm using an electronic balance, prior and after each test. The test samples after loading in the test rig were eroded for 1 min. at a given impingement angle and then weighed again to determine weight loss (Δw). The erosion rate (E_r) is then calculated by using the following equation:

$$E_r = \frac{\Delta w}{w_e} \qquad \text{..... (3.5)}$$

Where Δw is the mass loss of test sample in gm and w_e is the mass of eroding particles (i.e., testing time × particle feed rate). This procedure has been repeated until the erosion rate attains a constant steady-state value. In the present study the same procedure is repeated for 5 times (i.e. expose time was 5min).

The erosion efficiency (η) for the process was obtained by using the equation:

$$\eta = \frac{2E_r H}{\rho \times v^2} \qquad \text{..... (3.6)}$$

Where 'E_r' is erosion rate (kg/kg), 'H' is hardness of eroding material (MPa) and 'v' is velocity of impact (m/s), proposed by Sundararajan et al. [75]. Experimental results of the erosion test for different weight fraction of Rice-Husk fiber reinforced epoxy composites with different impingement angle and velocities are tabulated and presented in table-3.5 to 3.9.

3.12 RESULTS & DISCUSSION

Based on the tabulated results various graphs were plotted and presented in Figure-3.11 to 3.14 for different percentage of reinforcement under different test conditions.

Figure-3.11 to 3.14 illustrate the erosion wear rates of both neat epoxy and RH reinforced epoxy composite as a function of impingement angle under different impact velocities (48m/s to 109m/s). It is observed that Rice-Husk fiber epoxy composite of different % wt. fraction and also neat epoxy shows peak erosion rate (E_r max) at 45° and 60° impact angle and minimum erosion rate (E_r min) at normal incidence (90°) under all velocity of impact. It has been observed that for ductile material maximum erosion exists at low impact angles (15 ° to 30 °) whereas for brittle materials it is at normal angle (90°) [76]. Hence the maximum erosion occurring between 45° to 60° angles shows semi-ductile or semi-brittle material [77]. From the experimental results it is clear that RH fiber reinforced composites respond to solid particle impact neither behaves in a purely ductile nor in a purely brittle manner. This behavior can be termed as semi-brittle in nature. It is further observed that irrespective of impact velocity and impact angle, the erosion rate is highest for neat epoxy whereas erosion rate decreases to minimum as the % wt. fraction of fiber increases and the optimum value was found in 20% wt. fraction of Rice-Husk fiber reinforced epoxy composite.

The variation of steady-state erosion rate of all composite samples with impact velocity at different impact angles are shown in the form of a histogram in Figure-3.15 to 3.18. It can be observed from these histograms that erosion rate of all composite samples increases with increase in the impact velocity. However, 20% wt. fraction shows the least variation in the erosion rate with increase in the impact

velocity at low impact angle (α = 30°). Irrespective of impingement angle and impact velocity, there is a steady decrease in erosion rate with increase in fiber content has also been observed. This indicates that the erosion rate of composites is decreased only by the increased weight fraction of fiber content.

In the solid particle impact experiments the impact velocity of the erosive particles has a very strong effect on erosion rate. For any material, once steady state conditions have reached, the erosion rate 'E_r' can be expressed as a simple power function of impact velocity (v) [64]:

$$E_r = kv^n \quad \text{..... (3.7)}$$

where k is the constant of proportionality includes the effect of all the other variables and 'n' is the velocity exponent. The value of 'n' and 'k' are found by least-square fit ing of the data points in plots which represent the erosion rate dependence on impact velocity by using the power law. The value of 'n' is typically between 2 and 4, although much higher exponent is seen under some circumstances [68]. According to Pool et al. [64], for polymer composites behaving in ductile manner, the velocity exponent 'n' varies in the range 2-3 while for polymer composites behaving in brittle fashion the value of 'n' should be in the range of 3-5. Figure-3.19 to 3.22 illustrates the variation of erosion rate with impact velocities at different impingement angle for neat epoxy and its composites. The least-square fits to data point were obtained by using power law and the values of 'n' and 'k' are summarized in Table-3.6. The velocity exponents found for 30°, 45°, 60° and 90° impingement angles are in the range of 2.56-2.91, 2.53-2.70, 2.94-3.11 and 2.91-3.05 respectively. The value of velocity exponent 'n' shows that Rice-Husk filled epoxy composite behaves in a semi-brittle manner. This velocity exponent at various impingement angles are in conformity with Harsha et al. [78].

It has been reported by Sundararajan et al. [68, 75] that the erosion efficiency (η), can be used to characterize the nature and mechanism of erosion. They also showed that the ductile material possesses very low erosion efficiency i.e. $\eta << 100\%$, whereas the brittle material exhibits an erosion efficiency even greater than 100%. The values of erosion efficiencies of composites under this study are calculated using equation-3.2 and are listed in Table-3.11 & 3.12 along with their hardness values and operating

conditions. According to the categorization made by Roy et al. [68], it has been observed that the erosion efficiencies of Rice-Husk fiber epoxy composite varies from 1% to 5% for different impact velocities, indicating a semi-brittle erosion response. Further it is noticed that the erosion efficiency of all tests sample slightly increases with increase in impact velocity. Thus it can be conclude that the erosion efficiency is not exclusively a material property; but also depends on other operational variables such as impact velocity and impingement angle. The data shown in Table-3.11 & 3.12 are also indicates that the erosion efficiency of Rice-Husk fiber epoxy composite increase with increase in fiber content whereas the neat epoxy exhibits a lower value under all testing condition. This lower erosion efficiency of neat epoxy indicates a less erosion resistance in comparison to Rice-Husk fiber epoxy composite.

3.13 CONCLUSIONS

Based on the experimental results of mechanical test and abrasive wear test of rice husk fiber reinforced epoxy composite, the following conclusions has been drawn.

1. The Plain Rice-Husk (PRH) fiber can successfully be used as reinforcing agent to fabricate composite by suitably bonding with epoxy resin.
2. With increasing rice husk fibers reinforcement the tensile strength and flexural strength increases gradually and it is found that 15% weight fraction of Plain Rice-Husk reinforcement gives better strength than others.
3. Fiber breakages are found to be the predominant mode of failure as ascertained from the morphology of the treated fiber composites.
4. The incorporation of rice husk in to epoxy can significantly reduce the erosive wear loss. The optimal wear resistance property was obtained at a fiber content of 20% weight fraction.
5. The influence of impingement angle on erosive wear of composites under consideration exhibits semi-brittle erosive wear behavior with maximum wear rate between 45°-60° impingement angle.
6. With increasing of the velocity of impingement, erosion rate gradually increases.

7. In PRH-epoxy composites the erosion rate (E_r) displays power law behavior with particle velocity (v), $Er \alpha v^n$, where 'n' varies from 2.53 to 3.11 which further explains its semi-brittle behavior.
8. The erosion efficiency (η) values obtained experimentally also indicate that the Plain Rice-Husk (PRH) fiber reinforced epoxy composites exhibit semi-brittle erosion response (1.496%-5.442%).
9. The morphologies of eroded surface of the samples observed by SEM indicate that, material removal is mainly due to micro-cutting and micro-ploughing.

Table-3.3: Particle velocity under different pressure.

S. No.	Air Pressure (Bar)	Particle velocity (m/s)
1	1	48
2	2	70
3	3	82
4	4	109

Table-3.4: Experimental parameters for the erosion test.

Erodent	Silica sand
Erodent size (µm)	300-600
Impingement angle(α°)	30, 45, 60, 90
Impact velocity (m/s)	48, 70, 82, 109
Erodent feed rate (gm/min)	11
Test temperature	Room temperature
Standoff distance (mm)	10
Nozzle diameter (mm)	4
Time of experiment (min)	5

Table- 3.5: Cumulative weight loss & erosion rate of neat epoxy for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Neat Epoxy	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.011	2.056
	45	55	0.013	2.340
	60	55	0.011	1.929
	90	55	0.008	1.471
70	30	55	0.029	5.345
	45	55	0.048	8.800
	60	55	0.044	7.964
	90	55	0.027	4.836
82	30	55	0.049	8.964
	45	55	0.068	12.364
	60	55	0.074	13.455
	90	55	0.059	10.709
109	30	55	0.089	16.255
	45	55	0.108	19.636
	60	55	0.113	20.455
	90	55	0.090	16.364

Table- 3.6: Cumulative weight loss & erosion rate of 5% PRH for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	PRH 5%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.010	1.853
	45	55	0.012	2.240
	60	55	0.009	1.700
	90	55	0.007	1.309
70	30	55	0.020	3.636
	45	55	0.045	8.255
	60	55	0.037	6.764
	90	55	0.024	4.450
82	30	55	0.044	8.000
	45	55	0.063	11.436
	60	55	0.071	12.891
	90	55	0.051	9.200
109	30	55	0.083	15.000
	45	55	0.100	18.182
	60	55	0.108	19.564
	90	55	0.082	14.891

Table- 3.7: Cumulative weight loss & erosion rate of 10% PRH for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	PRH 10%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.008	1.469
	45	55	0.012	2.151
	60	55	0.009	1.600
	90	55	0.007	1.335
70	30	55	0.025	4.491
	45	55	0.039	7.109
	60	55	0.035	6.309
	90	55	0.021	3.782
82	30	55	0.043	7.727
	45	55	0.060	10.836
	60	55	0.066	11.945
	90	55	0.048	8.727
109	30	55	0.080	14.455
	45	55	0.095	17.291
	60	55	0.103	18.764
	90	55	0.076	13.873

Table- 3.8: Cumulative weight loss & erosion rate of 15% PRH for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	PRH 15%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.009	1.633
	45	55	0.011	2.007
	60	55	0.008	1.480
	90	55	0.006	1.164
70	30	55	0.017	3.145
	45	55	0.036	6.618
	60	55	0.031	5.636
	90	55	0.021	3.782
82	30	55	0.035	6.327
	45	55	0.052	9.400
	60	55	0.058	10.545
	90	55	0.042	7.600
109	30	55	0.075	13.636
	45	55	0.086	15.673
	60	55	0.097	17.618
	90	55	0.073	13.182

Table- 3.9: Cumulative weight loss & erosion rate of 20% PRH for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	PRH 20%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.006	1.169
	45	55	0.009	1.562
	60	55	0.007	1.336
	90	55	0.007	1.229
70	30	55	0.018	3.273
	45	55	0.033	5.927
	60	55	0.028	5.091
	90	55	0.019	3.364
82	30	55	0.031	5.600
	45	55	0.045	8.127
	60	55	0.052	9.527
	90	55	0.040	7.255
109	30	55	0.069	12.545
	45	55	0.078	14.127
	60	55	0.089	16.218
	90	55	0.068	12.418

Table- 3.10: Parameters characterizing the velocity dependence of erosion rate of Neat Epoxy & PRH reinforced composite.

Fiber Type	Angle (Degree)	k x 10^{-6}	n
Neat Epoxy	**30**	106	2.56
	45	107	2.62
	60	25	2.94
	90	12	3.04
PRH 5%	**30**	66	2.62
	45	121	2.58
	60	13	3.06
	90	10	3.05
PRH 10%	**30**	27	2.83
	45	127	2.53
	60	12	3.08
	90	14	2.96
PRH 15%	**30**	55	2.63
	45	111	2.58
	60	10	3.09
	90	9	3.03
PRH 20%	**30**	14	2.91
	45	52	2.70
	60	8	3.11
	90	16	2.91

Table-3.11: Erosion efficiency (η) of neat epoxy samples.

Impact Velocity (m/s)	Impact Angle (Degree)	Neat Epoxy H = 117.7 (MPa) ρ =1140 (Kg/m^3)
48	30	1.843
	45	2.097
	60	1.729
	90	1.318
70	30	2.253
	45	3.708
	60	3.356
	90	2.038
82	30	2.753
	45	3.797
	60	4.132
	90	3.289
109	30	2.825
	45	3.413
	60	3.555
	90	2.844

Table-3.12: Erosion efficiency (η) of PRH composite samples.

Impact Velocity (m/s)	Impact Angle (Degree)	PRH 5%	PRH 10%	PRH 15%	PRH 20%
		H=149.1 (MPa)	H=159.9 (MPa)	H=167.7 (MPa)	H=181.4 (MPa)
		ρ =1106 (Kg/m^3)	ρ =1044 (Kg/m^3)	ρ =1132 (Kg/m^3)	ρ =1026 (Kg/m^3)
48	**30**	2.168	1.953	2.100	1.794
	45	2.621	2.860	2.581	2.397
	60	1.989	2.127	1.903	2.051
	90	1.532	1.774	1.496	1.886
70	**30**	2.001	2.807	1.902	2.362
	45	4.542	4.444	4.002	4.277
	60	3.722	3.944	3.408	3.674
	90	2.449	2.364	2.287	2.427
82	**30**	3.208	3.520	2.788	2.945
	45	4.586	4.937	4.142	4.274
	60	5.169	5.442	4.647	5.010
	90	3.689	3.976	3.349	3.815
109	**30**	3.404	3.727	3.401	3.734
	45	4.126	4.458	3.908	4.205
	60	4.440	4.838	4.394	4.827
	90	3.379	3.577	3.287	3.696

Figure-3.10: Details of erosion test rig.

(1) Sand hopper (2) Conveyor belt system for sand flow (3) Pressure transducer, (4) Particle-air mixing chamber, (5) Nozzle, (6) X–Y and h axes assembly, (7) Sample holder.

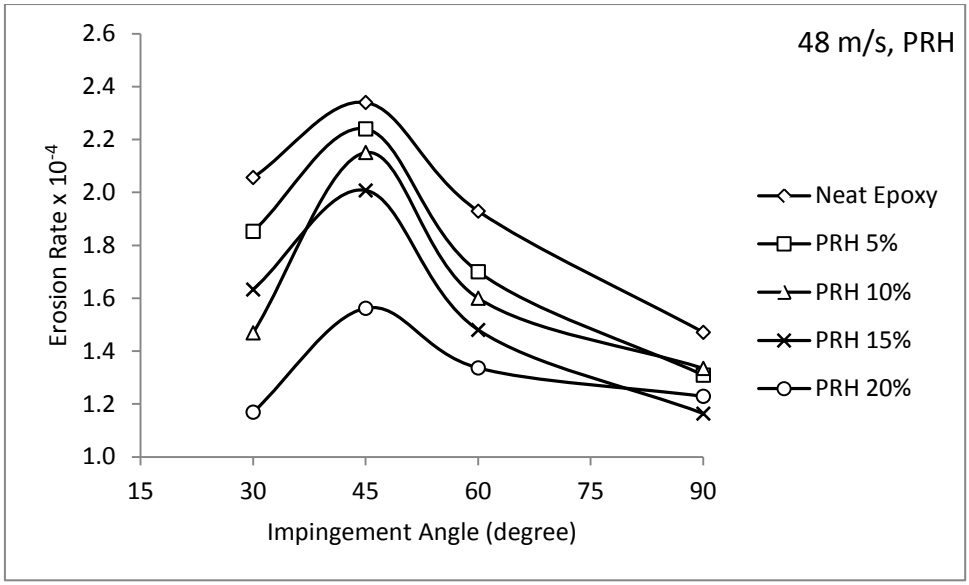


Figure-3.11: Variation of erosion rate with impingement angle of various Plain Rice-Husk epoxy composite at impact velocity of 48 m/s.

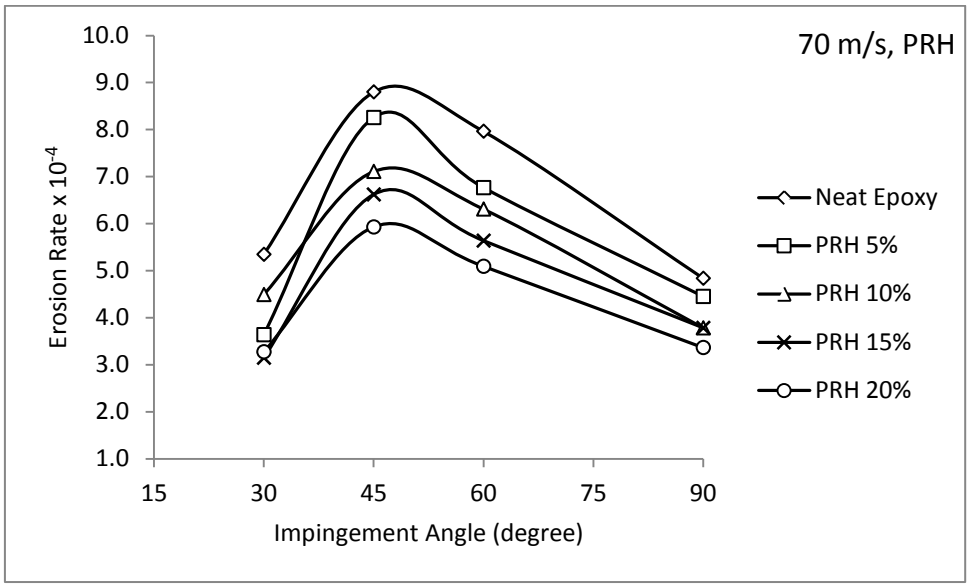


Figure-3.12: Variation of erosion rate with impingement angle of Epoxy & PRH-epoxy composite at impact velocity of 70 m/s.

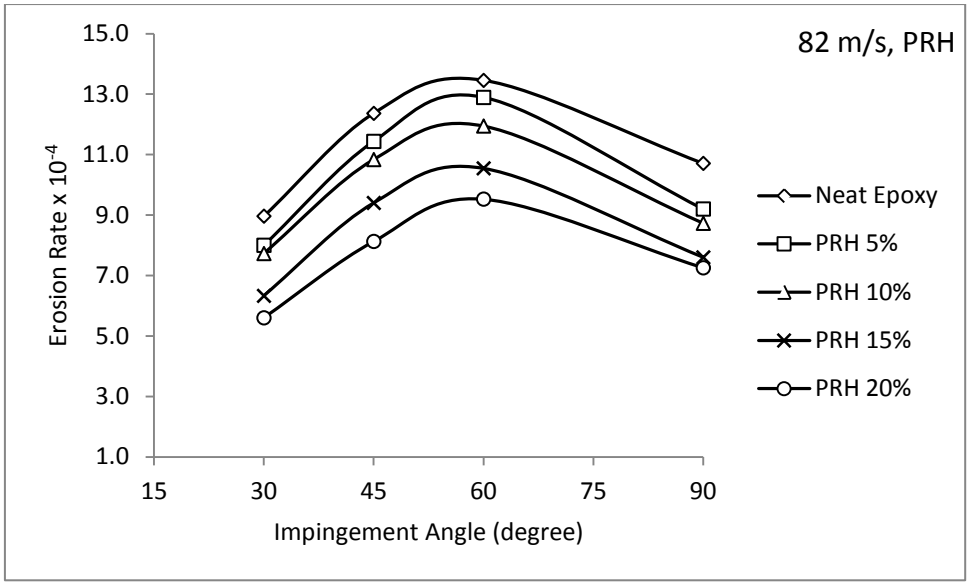


Figure-3.13: Variation of erosion rate with impingement angle of Epoxy & PRH-epoxy composite at impact velocity of 82 m/s.

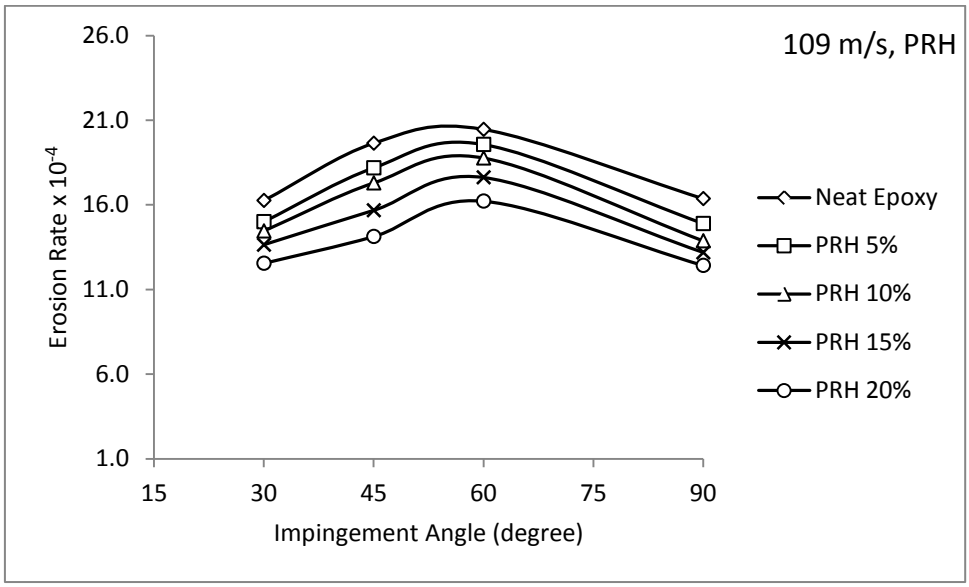


Figure-3.14: Variation of erosion rate with impingement angle of Epoxy & PRH-epoxy composite at impact velocity of 109 m/s.

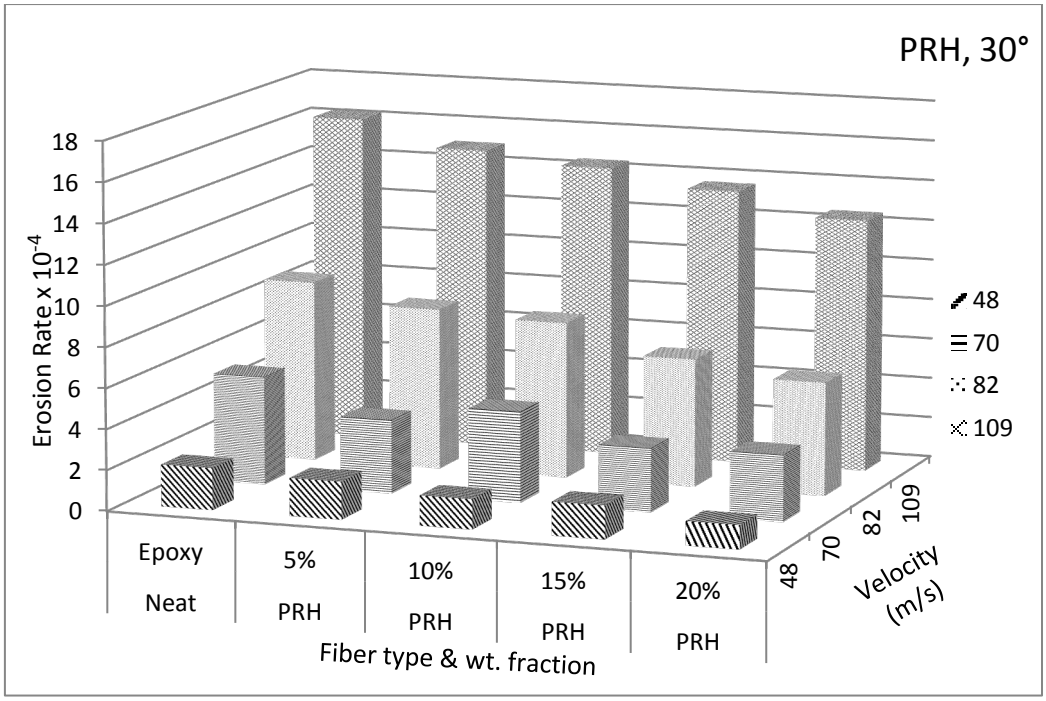


Figure-3.15: Histogram showing the steady state erosive wear rates of all the composites of PRH & epoxy at four impact velocities (i.e. at 48, 70, 82 and 109 m/s) for 30° impact angle.

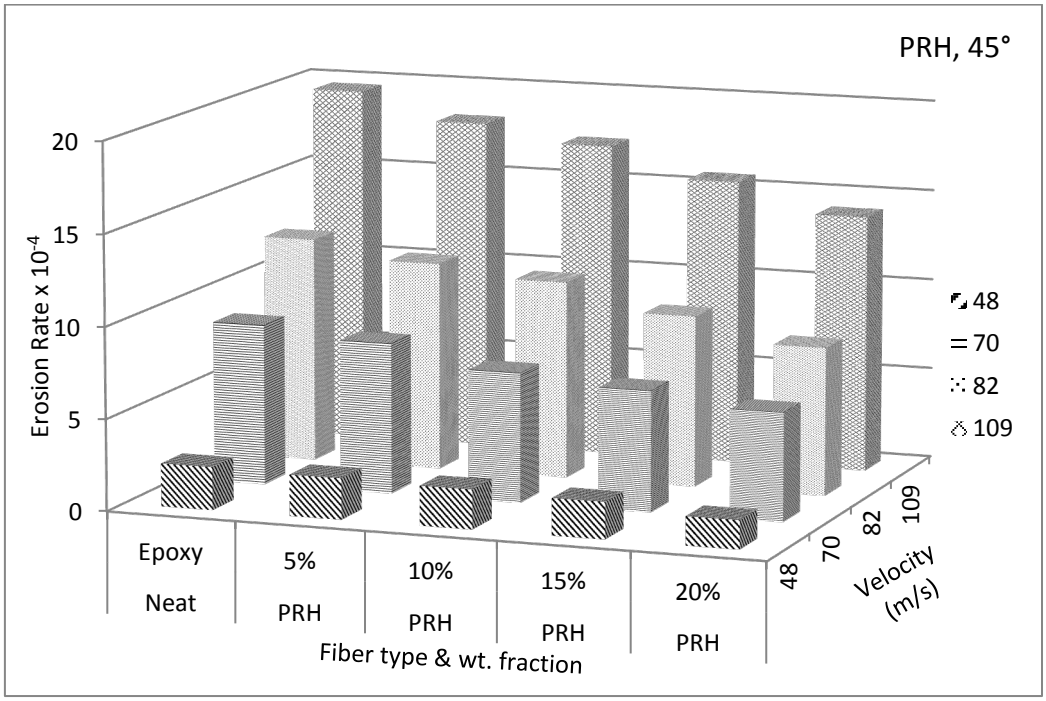


Figure-3.16: Histogram showing the steady state erosive wear rates of all the composites of PRH & epoxy at four impact velocities (i.e. at 48, 70, 82 and 109 m/s) for 45° impact angle.

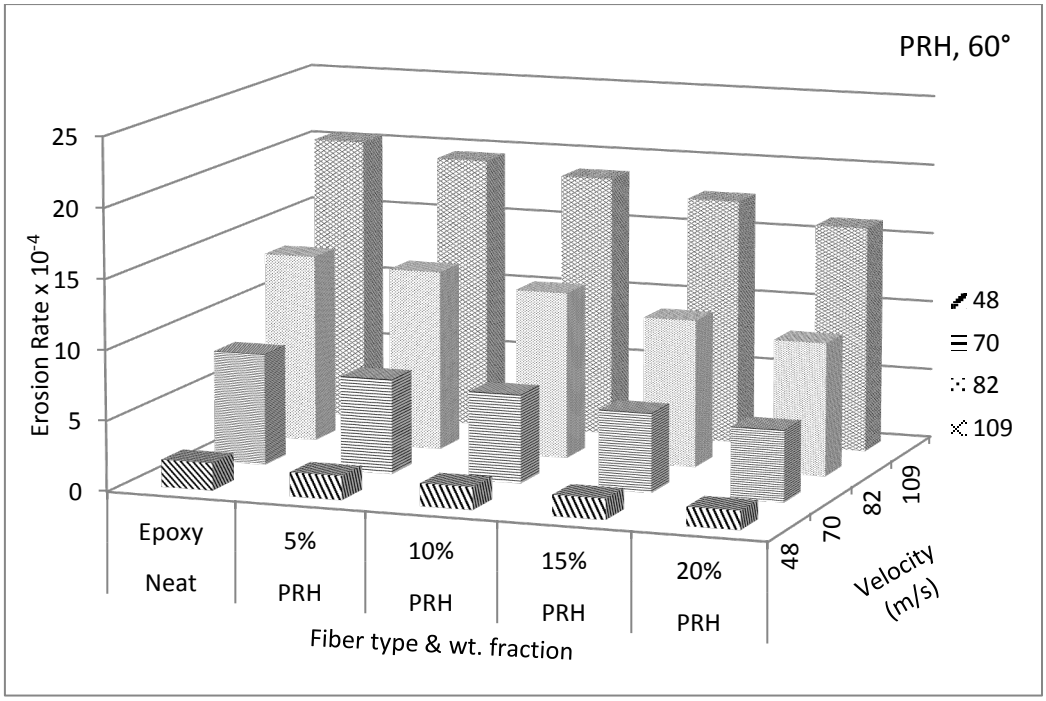


Figure-3.17: Histogram showing the steady state erosive wear rates of all the composites of PRH & epoxy at four impact velocities (i.e. at 48, 70, 82 and 109 m/s) for 60° impact angle.

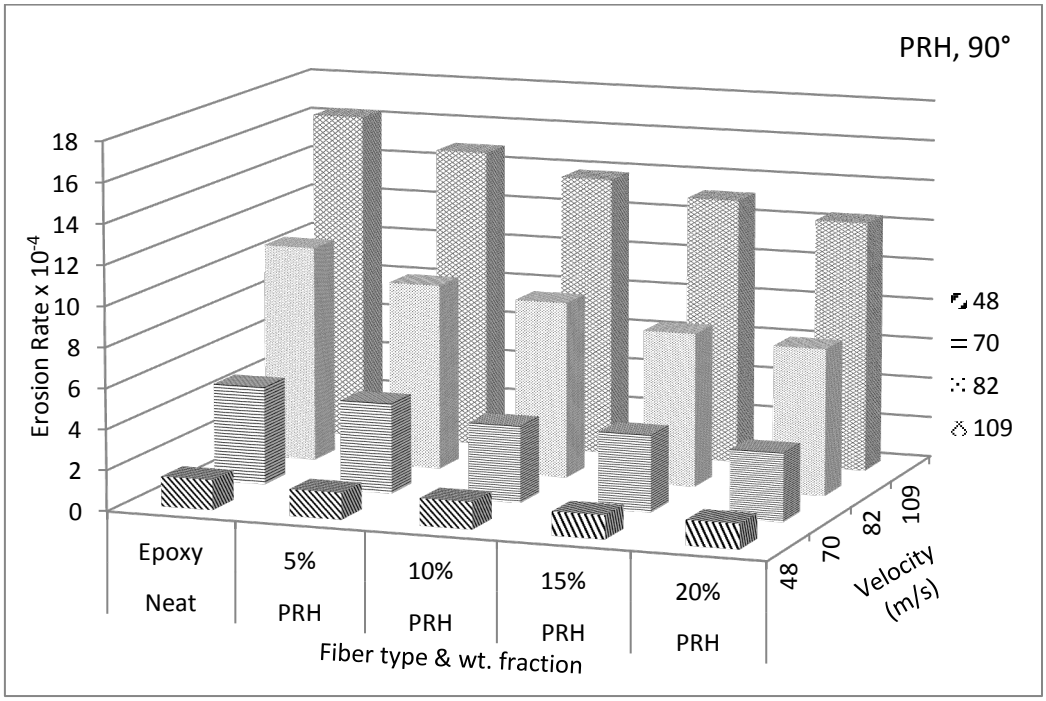


Figure-3.18: Histogram showing the steady state erosive wear rates of all the composites of PRH & epoxy at four impact velocities (i.e. at 48, 70, 82 and 109 m/s) for 60° impact angle.

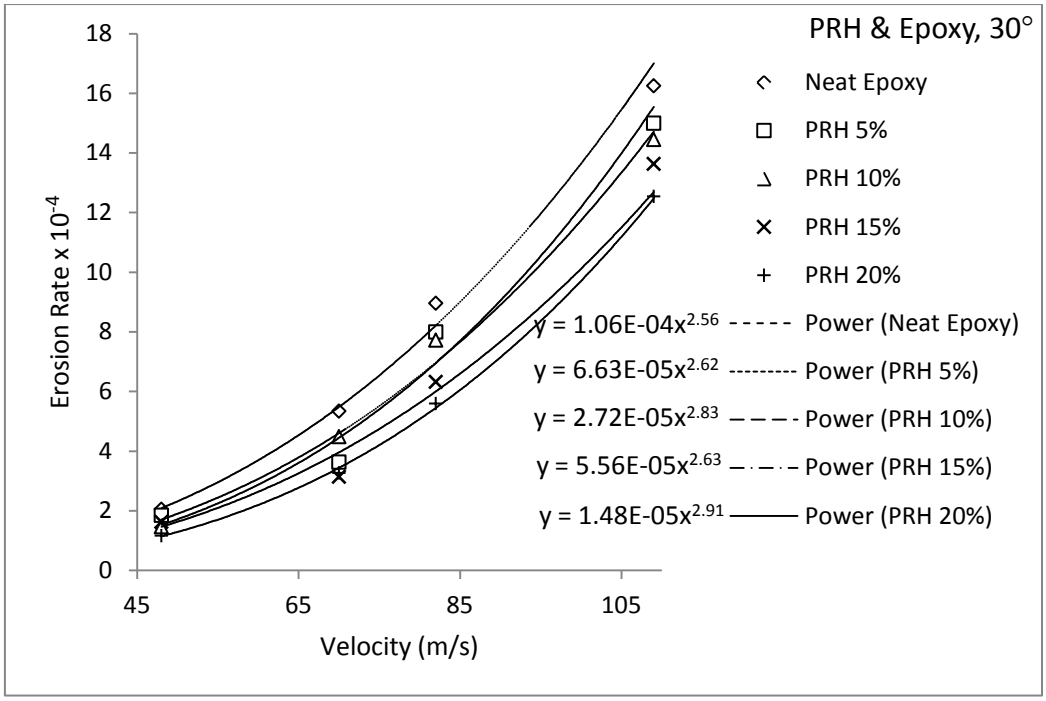


Figure-3.19: Variation of steady-state erosion rate of neat epoxy and PRH-epoxy composites as a function of impact velocity for 30° impact angle.

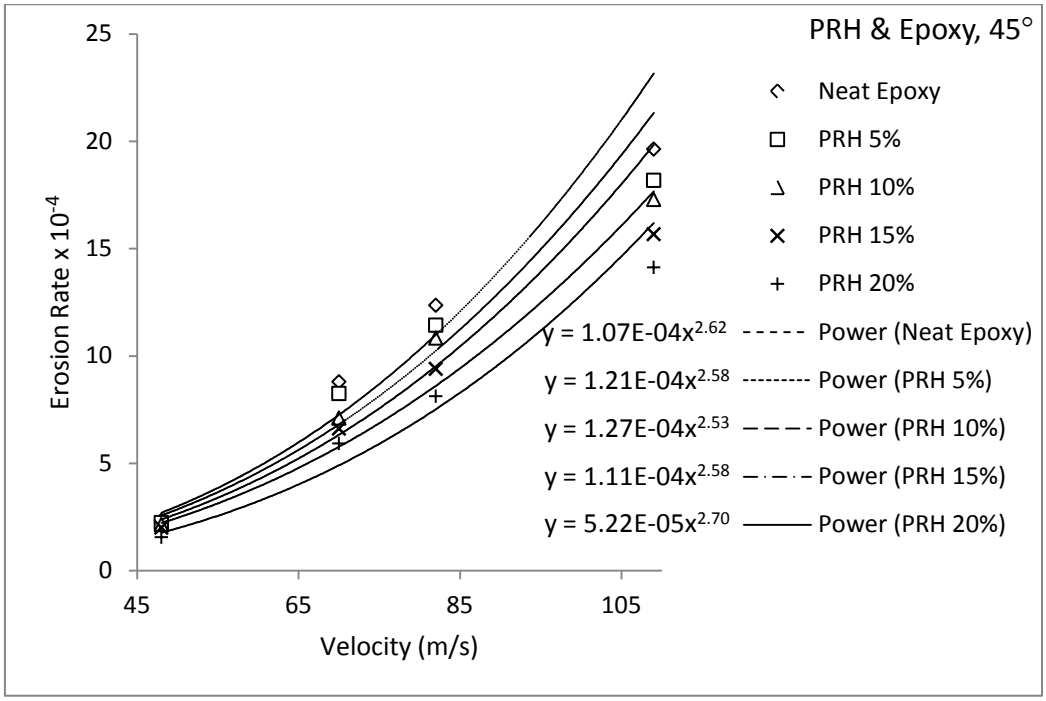


Figure-3.20: Variation of steady-state erosion rate of neat epoxy and PRH-epoxy composites as a function of impact velocity for 45° impact angle.

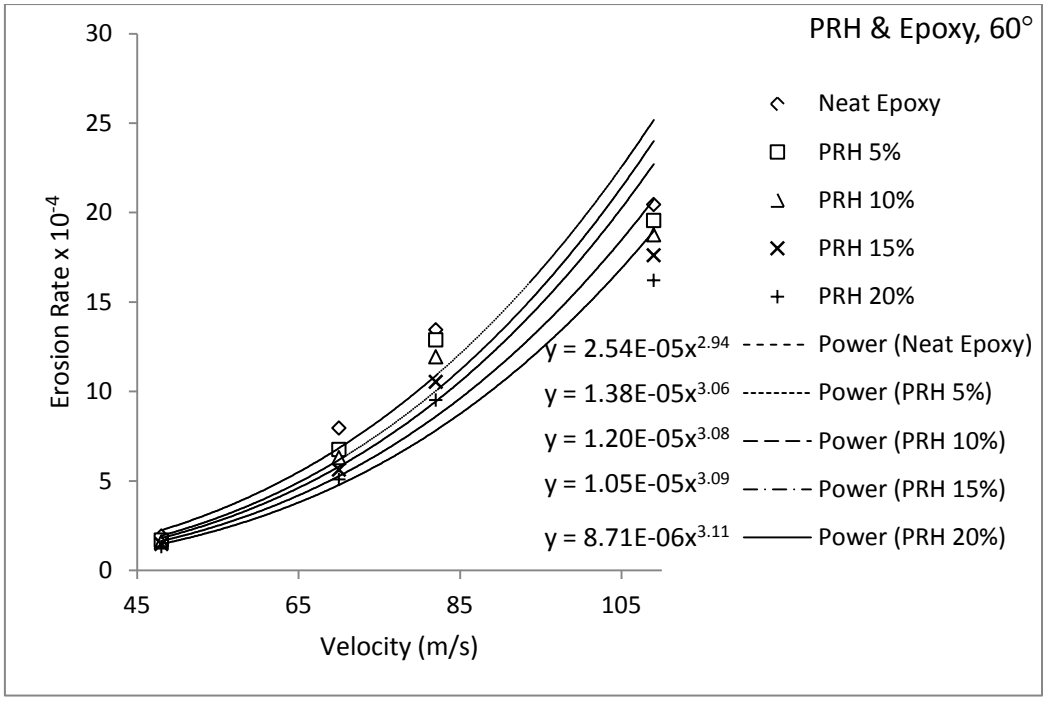


Figure-3.21: Variation of steady-state erosion rate of neat epoxy and PRH-epoxy composites as a function of impact velocity for 60° impact angle.

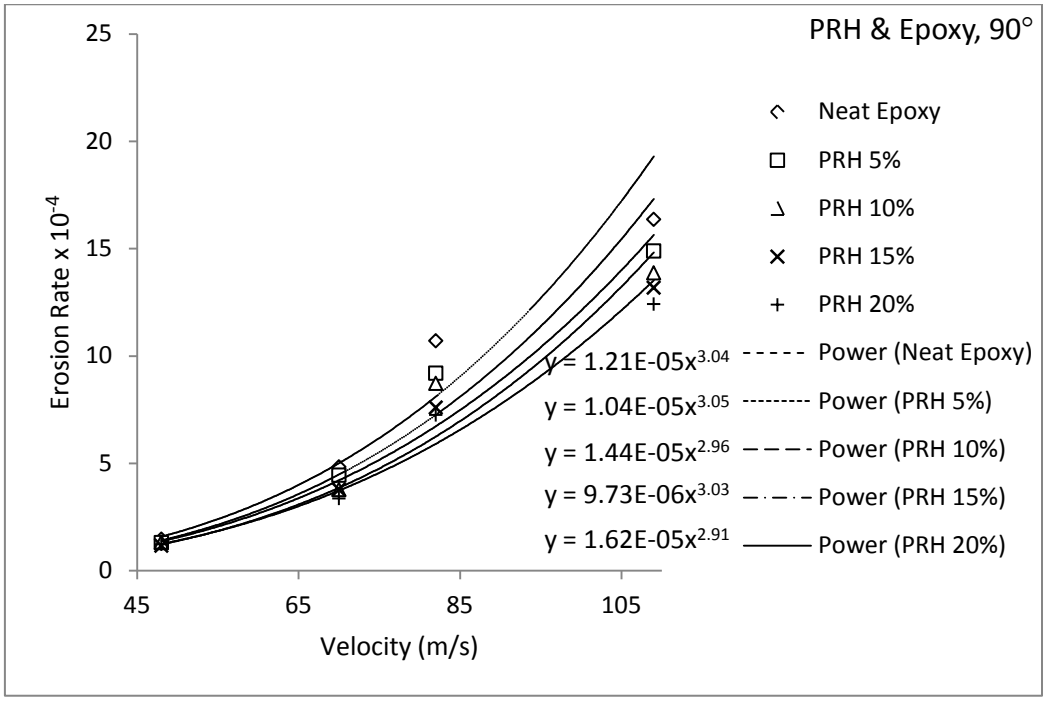


Figure-3.22: Variation of steady-state erosion rate of neat epoxy and PRH-epoxy composites as a function of impact velocity for 90° impact angle.

Chapter-4

4.1 INTRUDUCTION

In general natural fibers are hygroscopic in nature and they absorb or release moisture depending on environmental conditions. Amorphous cellulose and hemicellulose that present in the natural fiber are mostly responsible for the high moisture absorption, since they contain numerous easily accessible hydroxyl groups which give a high level of hydrophilic character to fiber. The high moisture absorption of the fiber occurs due to hydrogen bonding of water molecules to the hydroxyl groups within the fiber cell wall. This leads to a moisture build-up in the fiber cell wall (fiber swelling) and also in the fiber-matrix interface. This in turn becomes responsible for changes in the dimensions of cellulose-based composites, particularly in the thickness and the linear expansion due to reversible and irreversible swelling of the composites [60]. Another problem associated with fiber swelling is a reduction in the adhesion between the fiber and the matrix, leading to deterioration in the mechanical properties of the composite [61]. A good fiber-matrix bonding can decrease the rate and amount of moisture absorbed by the composite as well as improving the mechanical properties [79]. However in order to overcome this problem, chemical treatment has been considered as a good technique to reduce the hydroxyl group in the fibers.

4.2 CHEMICAL MODIFICATIONS OF FIBERS

The main disadvantages of natural fibers in composites are the poor compatibility between fiber and matrix and the relative high moisture absorption. Therefore a better understanding of the chemical composition and surface adhesive bonding of natural fiber is necessary for developing natural fiber reinforced composites.

The chemical treatment of natural fibers aimed at improving the adhesion between the fiber surface and the polymer matrix which can modify the fiber surface and also increases fiber strength. Moisture absorption of the composites can be reduced and their mechanical properties can be improved. In this experiment RH is chemically treated with Alkali and Acetone for better fiber and matrix interaction. Chemical treatment also helps to remove wax, silicon and other impurities [80].

4.3 METHODS OF CHEMICAL MODIFICATIONS

4.3.1 Acetone Treatment

Acetone treatment of natural fiber washes out some amount of cellulolignin, lignin and impurities of the fiber surface such as wax and dust content. Acetone is a non-polar organic solvent which usually dissolves the non-polar organic component hence increasing the fiber adhesion with the epoxy matrix.

The reaction involves the generation of acetic acid (CH3COOH) as by-product which must be removed from the lignocellulosic material before the fiber is used. Chemical modification with acetic anhydride (CH3-C(=O)-O-C(=O)-CH3) substitutes the polymer hydroxyl groups of the cell wall with acetyl groups, modifying the properties of these polymers so that they become hydrophobic . The reaction of acetic anhydride with fiber is shown as.

$$\textbf{Fiber-OH + } CH_3\textbf{-C (=O)-O-C (=O)-}CH_3 \rightarrow \textbf{Fiber-OCO}CH_3 + CH_3COOH$$

Acetylation can reduce the hygroscopic nature of natural fibers and increases the dimensional stability of composites. Acetylation was used in surface treatments of fiber for use in fiber-reinforced composites

4.3.2 Alkali Treatment

Alkali treatment of cellulosic fibres with sodium hydroxide (NaOH) is one of such methods that have been employed in order to improve the fiber–matrix interface bonding. This treatment removes wax, oils covering, some amount of lignin and the silicon content of the external surface of the RH fiber cell wall. It also depolymerizes cellulose and exposes it to the matrix.

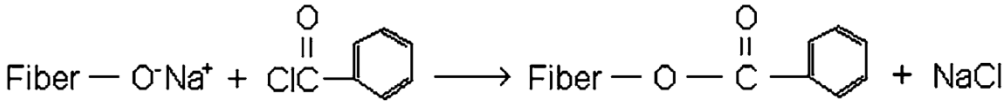
$$\textbf{Fiber – OH + NaOH} \rightarrow \textbf{Fiber – } O^- Na^+ + H_2O$$

It is reported that alkaline treatment has two effects on the natural fiber: (1) it increases surface roughness resulting in better mechanical interlocking; and (2) it increases the amount of cellulose exposed on the fiber surface to the matrix, which increases the number of possible reaction sites [81].

4.3.3 Benzoyl Chloride Treatment

Benzoyl chloride is most often used in fiber treatment. Benzoyl chloride includes benzoyl (C6H5C=O) which is attributed to the decreased hydrophilic nature of the treated fiber and improved interaction with the hydrophobic PS matrix. The reaction between the cellulosic hydroxyl group of the fiber and benzoyl chloride is given below:-

$$\textbf{Fiber} - \textbf{OH} + \textbf{NaOH} \rightarrow \textbf{Fiber} - \textbf{O}^{-}\ \textbf{Na}^{+} + \textbf{H}_2\textbf{O}$$



Benzoylation of fiber improves fiber matrix adhesion, thereby considerably increasing the strength of composite, decreasing its water absorption and improving its thermal stability.

The pre-treated fibers are suspended in 10% NaOH solution and agitated with benzoyl chloride. The mixture are kept for 15 min, filtered, washed thoroughly with water and dried between filter papers. The isolated fibers are then soaked in ethanol for 1 h to remove the benzoyl chloride and finally are washed with water and dried in the oven at 80° C for 24 h.

4.3.4 Silane Treatment

Silane is a chemical compound with chemical formula SiH4. Silanes are used as coupling agents to let glass fibers adhere to a polymer matrix, stabilizing the composite material. Silane coupling agents may reduce the number of cellulose hydroxyl groups in the fiber–matrix interface. In the presence of moisture, hydrolysable alkoxy group leads to the formation of silanols. The silanol then reacts with the hydroxyl group of the fiber, forming stable covalent bonds to the cell wall that are chemisorbed onto the fiber surface .Therefore, the hydrocarbon chains provided by the application of silane restrain the swelling of the fiber by creating a cross-linked network due to covalent bonding between the matrix and the fiber. The reaction schemes are given as follows:

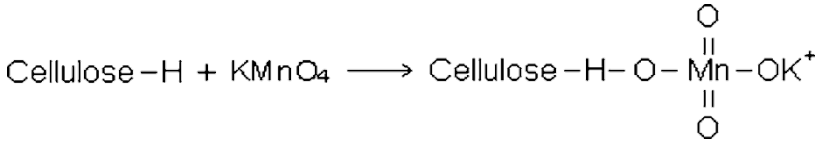
$$CH_2CHSi(OC2H5)_3 \xrightarrow{H_2O} CH_2CHSi(OH_3) + 3C_2H_5OH$$

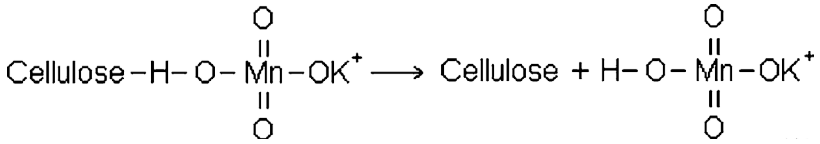
$$CH_2CHSi(OH_3) + Fiber\text{-}OH \rightarrow CH_2CHSi(OH)_2O\text{-}Fiber + H_2O$$

Silane coupling agents were also found to be effective in modifying natural fiber–polymer matrix interface and increasing the interfacial strength.

4.3.5 Permanganate Treatment

Permanganate is a compound that contains permanganate group MnO_4^-. Permanganate treatment leads to the formation of cellulose radical through MnO_3^- ion formation. Then, highly reactive Mn^{3+} ions are responsible for initiating graft copolymerization as given below:





Most permanganate treatments are conducted by using potassium permanganate (KMnO4) solution As a result of permanganate treatment, the hydrophilic tendency of the fibers is reduced, and hence, the water absorption of fiber-reinforced composite decreased.

4.3.6 Peroxide Treatment

In organic chemistry, peroxide is a specific functional group or a molecule with the functional group ROOR containing the divalent ion O−O. Organic peroxides tend to decompose easily to free radicals of the form RO; RO then reacts with the hydrogen group of the matrix and cellulose fibers. For example, the peroxide initiated free radical reaction between polyethylene (P.E.) matrix and cellulose fibers is shown by the following:

$$RO - OR \rightarrow 2RO$$

$$RO + PE - H \rightarrow ROH + PE$$

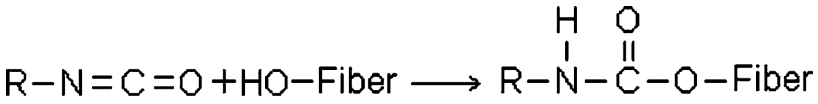
$$RO + Cellulose - H \rightarrow ROH + Cellulose$$

PE + Cellulose → PE – Cellulose

As a result of peroxide treatment, the hydrophilicity of the fibers decreased and the tensile properties increased.

4.3.7 Isocyanate Treatment

An isocyanate is a compound containing the isocyanate functional group –N=C=O, which is highly susceptible to reaction with the hydroxyl groups of cellulose and lignin in fibers. The reaction between fiber and isocyanate coupling agent is shown below:



where R could be different chemical groups (such as alkyl).

4.3.8 Acrylic Acid Treatment

Treatments like silane and acrylation led to strong covalent bond formation and thereby, the tensile strength and Young's modulus of treated fibers were improved marginally.

4.3.9Maleic anhydride Treatment

Maleated coupling agents are widely used to strengthen composites containing fillers and fiber reinforcements. The difference with other chemical treatments is that maleic anhydride is not only used to modify fiber surface but also the Polypropylene matrix to achieve better interfacial bonding and mechanical properties in composites.

4.3.10 Sodium chlorite ($NaClO_2$) Treatment

Sodium chlorite (NaClO2) usually is used in bleaching fibers. Tensile strength of bleached sisal fiber–polystyrene composite was less than other chemical treated fiber composites which may be due to the fact that delignification of the fiber lowered its tensile strength. But it was reported that flexural strength was better for bleached fiber composite because of lower stiffness and more flexible character of fibers after delignification. After delignification, the polymer replaces the role of lignin in fibers and makes composites more hydrophobic and tougher [82].

4.4 FABRICATION OF COMPOSITES WITH MODIFIED RICE HUSK AS REINFORCEMENT

For preparation of composites to conduct mechanical and erosion test the requirement of materials has been discussed in CHAPTER-3 Art: 3.5 & 3.6. It was found that for tensile and flexural test 15% weight fraction and for erosion test 20% weight fraction of fiber given the best result. To enhance the mechanical and erosion wear properties the reinforced material i.e. rice husk is modified with suitable chemical treatments. Out of the number of chemical modification method discussed in Art: 4.3, the two chemical modification methods i.e. acetone treatment and alkali treatment is adopted for present research work.

4.4.1 Fiber preparation with acetone treatment:

The rice husk fibers were washed in soxhlet extractor (Figure-4.1) with acetone for approximately 1.5-2.0 hrs. The acetone was evaporated (boiled at 63^0C) and condensed back into the volume with the fibers. Used acetone was discarded before the new batch was cleaned in the same manner. This process was repeated four times for each batch. The color of acetone changed after each batch of treatment due to the presence of waxes and organic materials. Hence in each batch the used acetone has to be changed. All the fibers were washed with pressurized water at a temperature of 90^0C for 70 minutes before acetone treatment. The fibers were then dried at room temperature for 24 hrs.

4.4.2 Fiber preparation with alkali treatment:

The rice husk fibers were soaked in a 5% (w/w) NaOH solution at room temperature, maintaining a liquor ratio of 15:1 (w/w). The fibers were kept immersed in the alkali solution for 4 hrs. as reported by Acharya et al. [83] that natural fibers soaked with alkali with 5% concentration and for 4 hours given the best result for the mechanical properties. The fibers were then washed several times with fresh water to remove any NaOH sticking to the fiber surface and if required washed with dilute acetic acid and again with water to achieve a final pH of 7. The fibers were then dried at room temperature for 48 hrs, followed by oven drying at 80°C for 8 hrs.

4.5 PREPARATION OF COMPOSITE SLABS FOR TENSILE TEST AND BEND TEST

The same procedure(i.e. as per CHAPTER-3, Art: 3.6 & 3.7) has been adopted to prepare composite slabs for tensile test and bend test .But only difference is that instead of plain (untreated) rice husk we are using chemically treated rice husk i.e. acetone treated and alkali treated rice husk has taken as a reinforced material.

4.6 TENSILE TEST AND BEND TEST

The method of conduction of these tests has been discussed in CHAPTER-3, Art-3.8 &3.9. The results obtained from the tests are presented in Table-4.1 & Table-4.2.

4.7 RESULTS AND DISCUSSION:

The tensile strength results for various specimens were plotted in Figure-4.2 & Figure-4.3. From Fig.4.2 and Fig.4.3 it is observed that treatment of rice husk with acetone and alkali increases the bonding strength with epoxy, hence the tensile strength and flexural strength increases. It is seen that 15% weight fraction of acetone treated rice husk reinforced epoxy composites gives maximum tensile strength and bending strength than untreated and alkali treated.

Table-4.1 Tensile and flexural strength for acetone treated rice husk reinforced epoxy composites.

Weight fraction of Acetone treated RH	Tensile Strength (MPa)	Flexural strength (MPa)
0%	14.07	18.18
5%	17.18	21.01
10%	19.96	29.32
15%	20.61	33.07
20%	18.10	29.11

Table-4.2 Tensile and flexural strength for alkali treated rice husk reinforced epoxy composites.

Weight fraction of Alkali treated RH	Tensile Strength (MPa)	Flexural strength (MPa)
0%	14.07	18.18
5%	15.41	20.65
10%	18.74	22.60
15%	19.51	25.87
20%	16.70	19.29

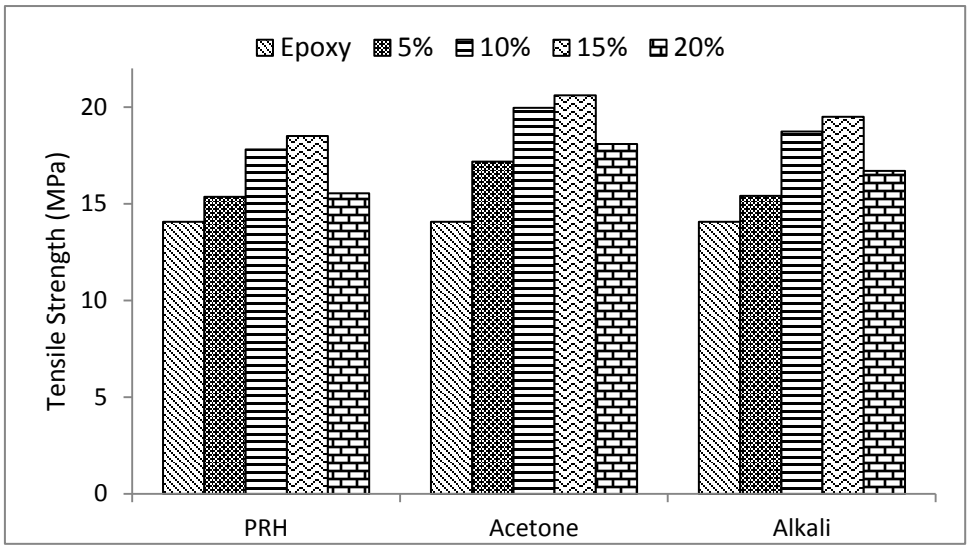


Figure-4.1 Effect of fiber modification by acetone and alkali treatment on tensile strength.

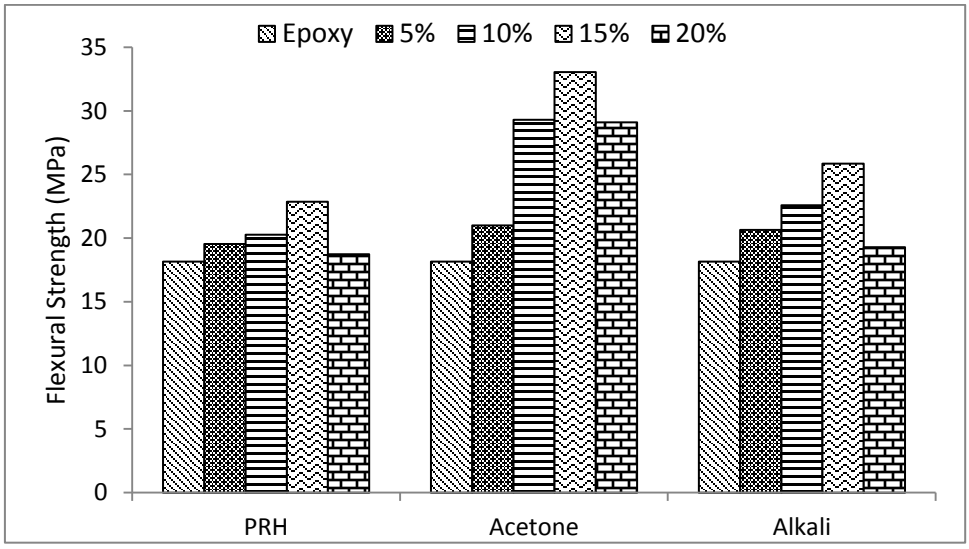


Figure-4.2 Effect of fiber modification by acetone and alkali treatment on flexural strength.

4.8 PREPARATION OF SPECIMENS FOR SOLID PARTICLE EROSION TEST

The same procedure (i.e. as per CHAPTER-3, Art: 3.6 & 3.11) has been adopted to prepare composite slabs for erosion tests. But only difference is that instead of plain (untreated) rice husk we are using chemically treated rice husk i.e. acetone treated and alkali treated rice husk has taken as a reinforced material.

4.9 CALCULATION

After the experiments the values of weight loss erosion rate velocity exponents & erosion efficiency have been calculated as per Chapter-3, Art- 3.11.2 and the values are listed in the tables 4.3 to table 4.13.

4.10 X-RAY DIFFRACTION

X-ray diffraction is a useful method for evaluating the crystallographic structure of semi-crystalline materials such as Rice-Husk fiber. A Philips X-ray diffractometer PAN alytical, employing CuKα ($\lambda = 1.54$) radiation and a graphite monochromator with a current of 40 mA and a voltage of 40 mV was used with a diffraction intensity in the range of 5° to 45° (2θ-angle range).

In Figure 4.28, one peak can be observed for PRH, alkali & acetone: at 22.7° 2θ, which indicates the presence of amorphous silica, quartz & calcite which corresponds to carbon (Krishnarao et al., 2001) [84]. Plane Rice-Husk shows highest crystallinity than treated rice husk (Acetone & Alkali) although important changes in the diffractogram were not observed after treatment. Due to treatment the peak was decreasing this shows that due to treatment some of the silica is washed out.

4.11 SCANNING ELECTRON MICROSCOPY STUDY

The morphology of the untreated and treated fiber surfaces has been studied using scanning electron microscope (SEM) JEOL JSM-6480LV. The sample surfaces were platinum coated to make them conductive prior to SEM observation. The eroded surfaces of RH-epoxy composites subjected to different impingement angles at different impact velocities are shown in Figure 4.29. The wear mechanisms in neat epoxy are more dominated by brittle failure. The erosion damage was dominantly reflected by formation of micro-cracks at surface which finally forms wear debris. Rice-Husk contains silica which is a typical brittle material. During normal

impingement greater amount of impact energy is utilized for crack initiation and local fracture of material. The pulverized wear debris is adhered to the matrix and helps in reducing further wear of composite. This effect is responsible for the low erosion rate of RH-composite as compared to neat epoxy.

4.12 RESULTS AND DISCUSSION

Fig-4.4 to Fig-4.11 shows the comparison of erosion wear rate between untreated and treated rice husk epoxy composites. It is clear from the figure that in case of treated rice husk epoxy composite the erosion wear rate is minimum as compared to untreated rice husk epoxy composite. This happened because the compatibility between rice husk particles and polymer increases due to fiber treatment. This is possible because the treatment completely wets the surface of RH and more and more OH groups are used for chemical bonding. From Fig-4.4 to Fig 4.11 it is observed that if the rice husk treated with acetone then it gives better erosive wear property than untreated and alkali treated rice husk reinforced epoxy composites.

4.13 CONCLUSION

1. Chemical treatment of RH fiber increased the fiber-matrix adhesion and in both kind of treatments i.e. acetone treatment and alkali treatment the mechanical properties of the composite improved.
2. Based on mechanical testing it was observed that acetone treatment of the RH fiber gives optimum result as compared to PRH and alkali treatment and 15% acetone treated RH-epoxy composite shown the best result.
3. Fiber breakages are found to be the predominant mode of failure as ascertained from the morphology of the treated fiber composites.
4. The incorporation of chemically treated rice husk in to epoxy reduced the erosive wear loss significantly compared to PRH epoxy composite.
5. Erosion test of the chemically treated RH epoxy composite shown that alkali treatment give maximum erosion resistance to the composite compared to the acetone treatment.
6. The optimal wear resistance property was obtained at a fiber content of 20% weight fraction with alkali treatment.

7. The influence of impingement angle on erosive wear of composites under consideration exhibits semi-brittle erosive wear behavior with maximum wear rate between 45°-60° impingement angle.
8. With increasing of the velocity of impingement, erosion rate gradually increases.
9. In chemically treated RH-epoxy composites the erosion rate (E_r) displayed the power law behavior with particle velocity (v), $Er \alpha v^n$, where 'n' varies from 2.40 to 3.19 which further explains its semi-brittle behavior.
10. The erosion efficiency (η) values obtained experimentally also indicate that the Plain Rice-Husk (PRH) fiber reinforced epoxy composites exhibit semi-brittle erosion response (1.211%-5.142%).
11. The morphologies of eroded surface of the samples observed by SEM indicate that, material removal is mainly due to micro-cutting and micro-ploughing.

Table- 4.3: Weight loss & erosion rate of 5% Acetone treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Acetone 5%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.010	1.753
	45	55	0.012	2.164
	60	55	0.010	1.805
	90	55	0.006	1.175
70	30	55	0.024	4.436
	45	55	0.045	8.164
	60	55	0.036	6.545
	90	55	0.023	4.182
82	30	55	0.041	7.382
	45	55	0.056	10.255
	60	55	0.068	12.364
	90	55	0.045	8.145
109	30	55	0.077	14.055
	45	55	0.094	17.055
	60	55	0.101	18.364
	90	55	0.079	14.400

Table- 4.4: Weight loss & erosion rate of 10% Acetone treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Acetone 10%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	**30**	**55**	0.009	1.725
	45	**55**	0.011	2.087
	60	**55**	0.009	1.549
	90	**55**	0.008	1.442
70	**30**	**55**	0.023	4.182
	45	**55**	0.037	6.691
	60	**55**	0.033	6.000
	90	**55**	0.020	3.636
82	**30**	**55**	0.039	7.091
	45	**55**	0.056	10.182
	60	**55**	0.063	11.527
	90	**55**	0.053	9.691
109	**30**	**55**	0.075	13.636
	45	**55**	0.090	16.400
	60	**55**	0.097	17.709
	90	**55**	0.085	15.527

Table- 4.5: Weight loss & erosion rate of 15% Acetone treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Acetone 15%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	**30**	**55**	0.009	1.573
	45	**55**	0.011	1.913
	60	**55**	0.008	1.373
	90	**55**	0.006	1.091
70	**30**	**55**	0.023	4.091
	45	**55**	0.034	6.091
	60	**55**	0.028	5.091
	90	**55**	0.023	4.182
82	**30**	**55**	0.033	6.000
	45	**55**	0.048	8.800
	60	**55**	0.054	9.891
	90	**55**	0.046	8.309
109	**30**	**55**	0.071	12.855
	45	**55**	0.080	14.545
	60	**55**	0.093	16.891
	90	**55**	0.077	14.055

Table- 4.6: Weight loss & erosion rate of 20% Acetone treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Acetone 20%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.006	1.053
	45	55	0.008	1.475
	60	55	0.007	1.218
	90	55	0.007	1.209
70	30	55	0.015	2.727
	45	55	0.028	5.145
	60	55	0.023	4.145
	90	55	0.016	2.896
82	30	55	0.028	5.000
	45	55	0.041	7.527
	60	55	0.049	8.836
	90	55	0.037	6.782
109	30	55	0.062	11.345
	45	55	0.067	12.255
	60	55	0.086	15.564
	90	55	0.073	13.327

Table- 4.7: Weight loss & erosion rate of 5% Alkali treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Alkali 5%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.008	1.515
	45	55	0.011	2.078
	60	55	0.008	1.527
	90	55	0.008	1.382
70	30	55	0.027	4.873
	45	55	0.040	7.273
	60	55	0.031	5.618
	90	55	0.023	4.182
82	30	55	0.040	7.273
	45	55	0.050	9.091
	60	55	0.063	11.527
	90	55	0.049	8.836
109	30	55	0.070	12.727
	45	55	0.088	16.000
	60	55	0.094	17.055
	90	55	0.074	13.527

Table- 4.8: Weight loss & erosion rate of 10% Alkali treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Alkali 10%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.008	1.424
	45	55	0.011	1.971
	60	55	0.008	1.442
	90	55	0.007	1.256
70	30	55	0.016	2.998
	45	55	0.033	6.036
	60	55	0.029	5.182
	90	55	0.018	3.200
82	30	55	0.035	6.273
	45	55	0.047	8.509
	60	55	0.060	10.818
	90	55	0.044	7.909
109	30	55	0.063	11.473
	45	55	0.080	14.545
	60	55	0.090	16.291
	90	55	0.064	11.636

Table- 4.9: Weight loss & erosion rate of 15% Alkali treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Alkali 15%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.008	1.402
	45	55	0.010	1.796
	60	55	0.007	1.253
	90	55	0.005	0.987
70	30	55	0.016	2.836
	45	55	0.028	5.091
	60	55	0.023	4.200
	90	55	0.019	3.418
82	30	55	0.025	4.600
	45	55	0.042	7.636
	60	55	0.050	9.164
	90	55	0.036	6.545
109	30	55	0.059	10.655
	45	55	0.069	12.600
	60	55	0.084	15.182
	90	55	0.066	12.018

Table- 4.10: Weight loss & erosion rate of 20% Alkali treated RH epoxy composite for a period of 5 minutes at different velocities and angles.

Velocity (m/s)	Angle (degree)	Weight of impingement (gm)	Alkali 20%	
			Weight loss (gm)	Erosion Rate $E_r \times 10^{-4}$ (gm/gm)
48	30	55	0.005	0.905
	45	55	0.007	1.335
	60	55	0.006	1.164
	90	55	0.006	1.104
70	30	55	0.013	2.331
	45	55	0.023	4.255
	60	55	0.019	3.473
	90	55	0.015	2.718
82	30	55	0.023	4.164
	45	55	0.035	6.436
	60	55	0.044	8.036
	90	55	0.033	6.000
109	30	55	0.051	9.273
	45	55	0.060	10.964
	60	55	0.078	14.236
	90	55	0.061	11.073

Table- 4.11: Parameters characterizing the velocity dependence of erosion rate of acetone and alkali treated RH reinforced epoxy composite.

Fiber Type	Angle (Degree)	k x 10^{-6}	n
Acetone 5%	**30**	85	2.57
	45	147	2.52
	60	25	2.91
	90	7	3.12
Acetone 10%	**30**	88	2.55
	45	120	2.55
	60	12	3.05
	90	11	3.03
Acetone 15%	**30**	79	2.55
	45	134	2.50
	60	8	3.13
	90	5	3.19
Acetone 20%	**30**	12	2.92
	45	67	2.61
	60	5	3.19
	90	10	3.01

Alkali 5%	**30**	65	2.62
	45	158	2.48
	60	13	3.04
	90	21	2.88
Alkali 10%	**30**	54	2.61
	45	162	2.45
	60	11	3.06
	90	21	2.84
Alkali 15%	**30**	90	2.47
	45	175	2.40
	60	7	3.14
	90	6	3.11
Alkali 20%	**30**	13	2.86
	45	63	2.60
	60	6	3.15
	90	14	2.89

Table-4.12: Erosion efficiency (η) of acetone treated RH reinforced epoxy composite.

Impact Velocity (m/s)	Impact Angle (Degree)	Acetone 5%	Acetone 10%	Acetone 15%	Acetone 20%
		H=136.3 (MPa)	H=144.2 (MPa)	H=153 1 (MPa)	H=162.8 (MPa)
		ρ=1148 (Kg/m^3)	ρ =1161 (Kg/m^3)	ρ =1133 (Kg/m^3)	ρ =1129 (Kg/m^3)
48	30	1.806	1.860	1.844	1.318
	45	2.230	2.250	2.242	1.846
	60	1.861	1.670	1.609	1.525
	90	1.211	1.555	1.279	1.513
70	30	2.150	2.120	2.255	1.605
	45	3.956	3.392	3.357	3.028
	60	3.172	3.042	2.806	2.440
	90	2.027	1.843	2.305	1.705
82	30	2.607	2.620	2.410	2.145
	45	3.621	3.761	3.535	3.228
	60	4.366	4.259	3.973	3.790
	90	2.877	3.580	3.337	2.909
109	30	2.809	2.851	2.922	2.754
	45	3.409	3.429	3.306	2.975
	60	3.670	3.703	3.840	3.778
	90	2.878	3.246	3.195	3.235

Table-4.13: Erosion efficiency (η) of alkali treated RH reinforced epoxy composite.

Impact Velocity (m/s)	Impact Angle (Degree)	Alkali 5%	Alkali 10%	Alkali 15%	Alkali 20%
		H=168.7 (MPa)	H=177.5 (MPa)	H=203 (MPa)	H=194.2 (MPa)
		ρ=1125 (Kg/m^3)	ρ =1131 (Kg/m^3)	ρ =1131 (Kg/m^3)	ρ =1133 (Kg/m^3)
48	30	1.971	1.939	2.184	1.347
	45	2.705	2.685	2.799	1.986
	60	1.988	1.964	1.952	1.731
	90	1.799	1.712	1.538	1.642
70	30	2.982	1.920	2.078	1.631
	45	4.451	3.867	3.730	2.977
	60	3.439	3.319	3.077	2.430
	90	2.560	2.050	2.504	1.902
82	30	3.244	2.928	2.456	2.123
	45	4.055	3.972	4.077	3.281
	60	5.142	5.050	4.892	4.097
	90	3.941	3.692	3.494	3.059
109	30	3.213	3.031	3.219	2.675
	45	4.039	3.843	3.807	3.163
	60	4.305	4.304	4.587	4.108
	90	3.415	3.074	3.631	3.195

Figure 4.3: Soxhlet Extractor

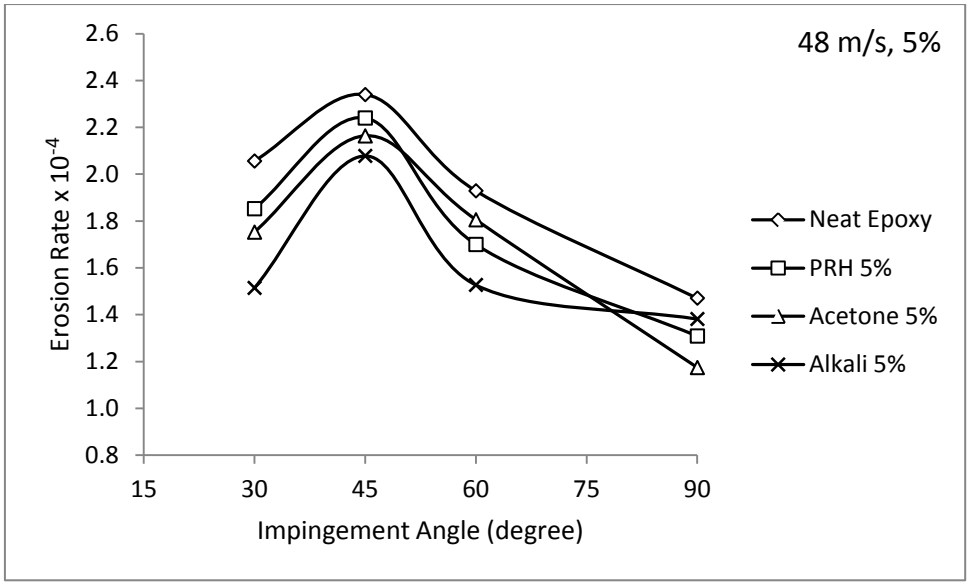


Figure-4.4: Comparison of erosion rate versus impingement angle of neat epoxy & 5% wt. fraction of PRH and treated RH epoxy composite at the velocity of 48 m/s.

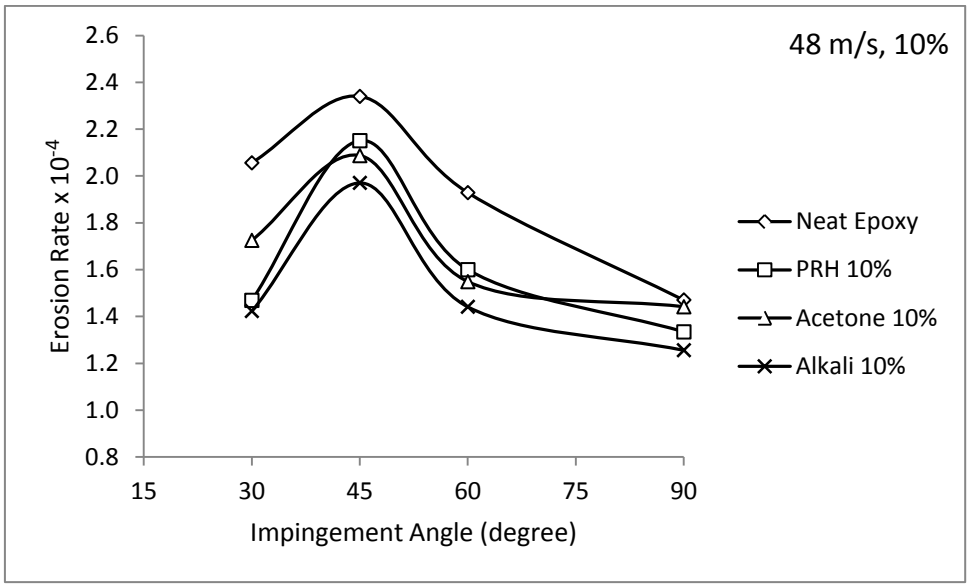


Figure-4.5: Comparison of erosion rate versus impingement angle of neat epoxy & 10% wt. fraction of PRH and treated RH epoxy composite at the velocity of 48 m/s.

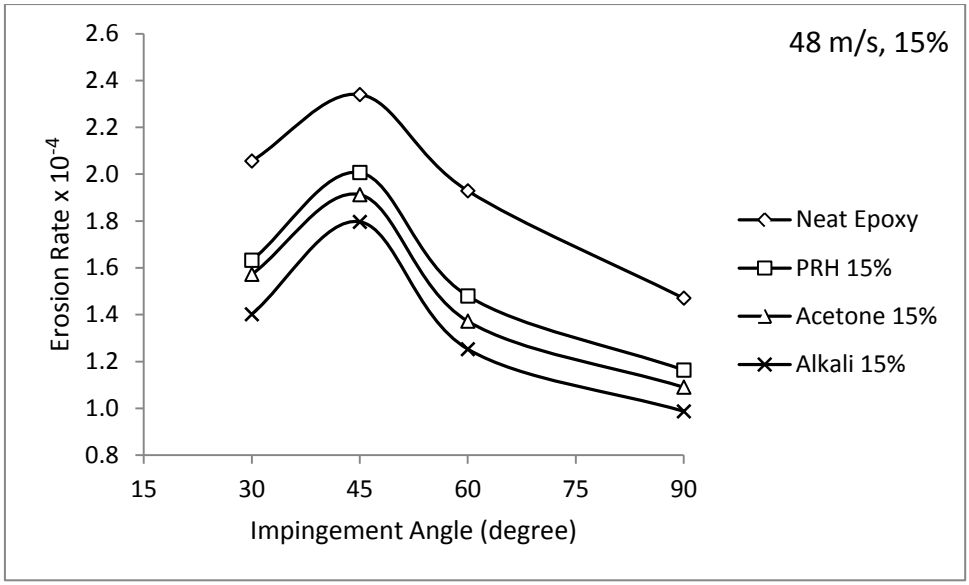


Figure-4.6: Comparison of erosion rate versus impingement angle of neat epoxy & 15% wt. fraction of PRH and treated RH epoxy composite at the velocity of 48 m/s.

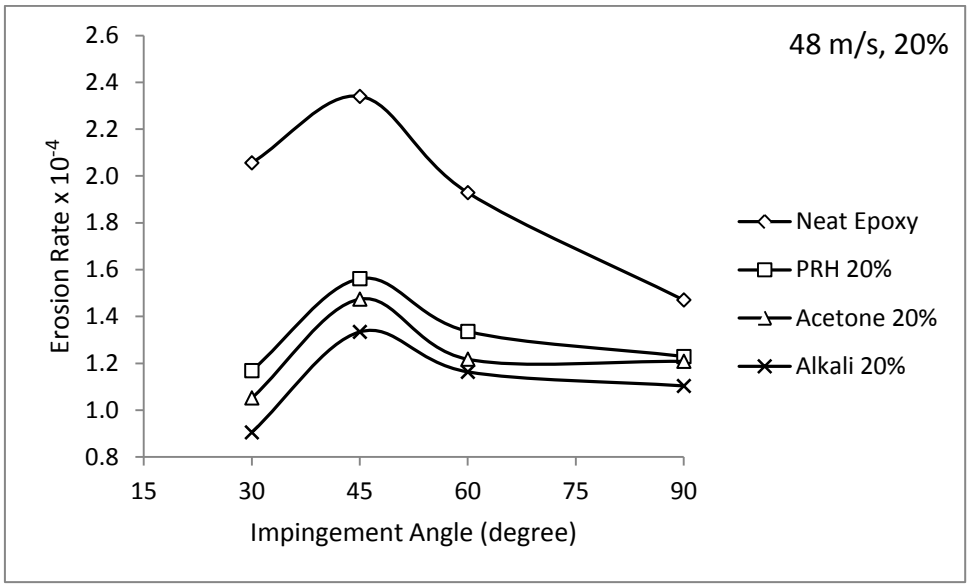


Figure-4.7: Comparison of erosion rate versus impingement angle of neat epoxy & 20% wt. fraction of PRH and treated RH epoxy composite at the velocity of 48 m/s.

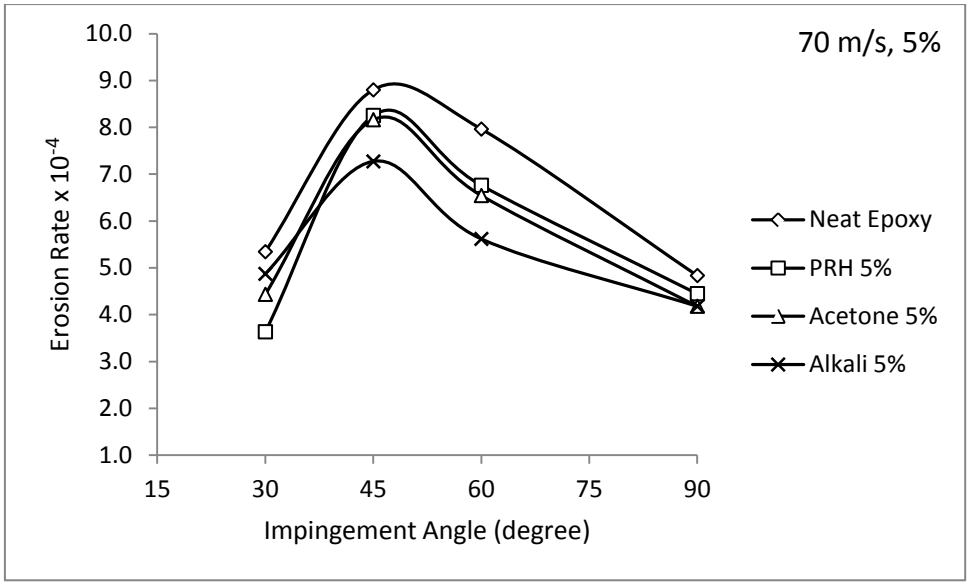


Figure-4.8: Comparison of erosion rate versus impingement angle of neat epoxy & 5% wt. fraction of PRH and treated RH epoxy composite at the velocity of 70 m/s.

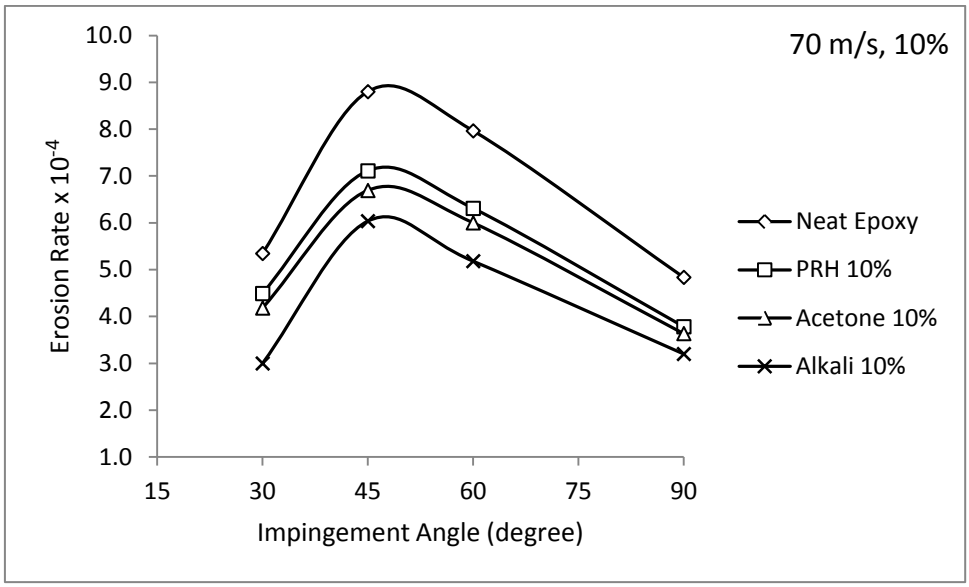


Figure-4.9: Comparison of erosion rate versus impingement angle of neat epoxy & 10% wt. fraction of PRH and treated RH epoxy composite at the velocity of 70 m/s.

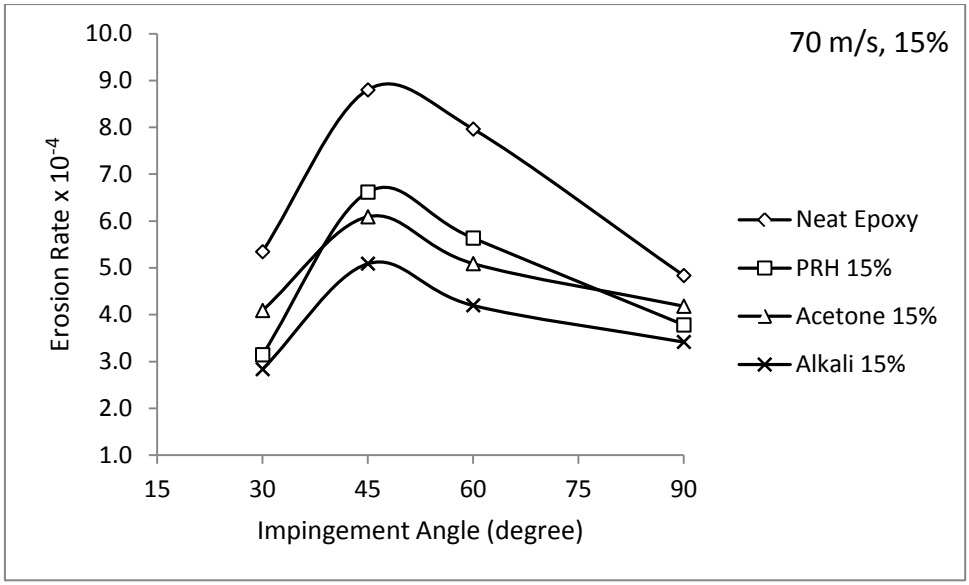


Figure-4.10: Comparison of erosion rate versus impingement angle of neat epoxy & 15% wt. fraction of PRH and treated RH epoxy composite at the velocity of 70 m/s.

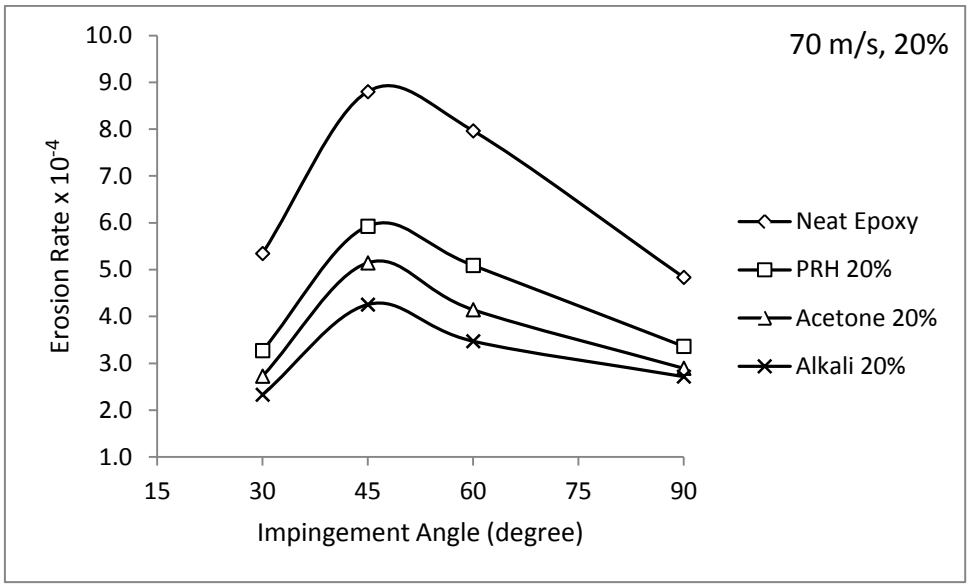


Figure-4.11: Comparison of erosion rate versus impingement angle of neat epoxy & 20% wt. fraction of PRH and treated RH epoxy composite at the velocity of 70 m/s.

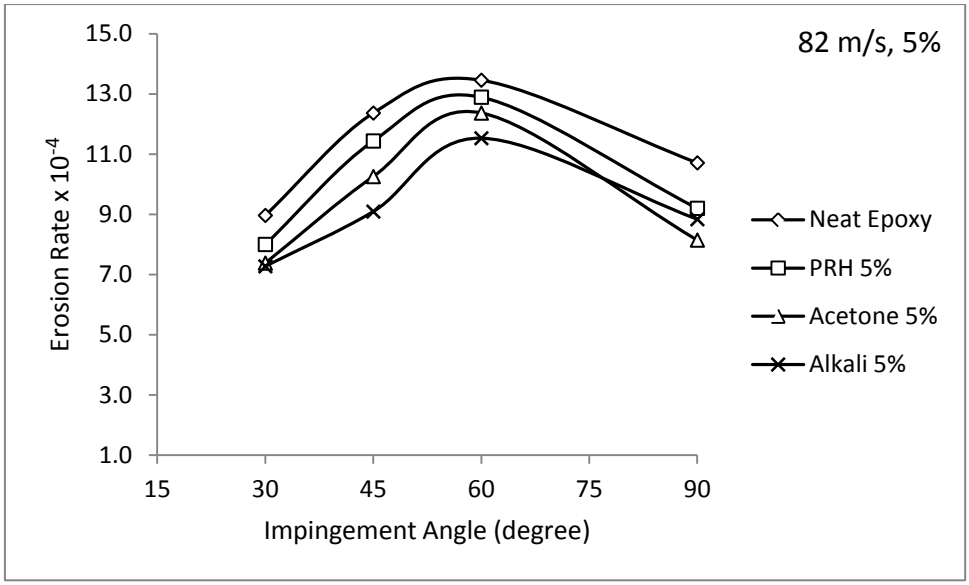


Figure-4.12: Comparison of erosion rate versus impingement angle of neat epoxy & 5% wt. fraction of PRH and treated RH epoxy composite at the velocity of 82 m/s.

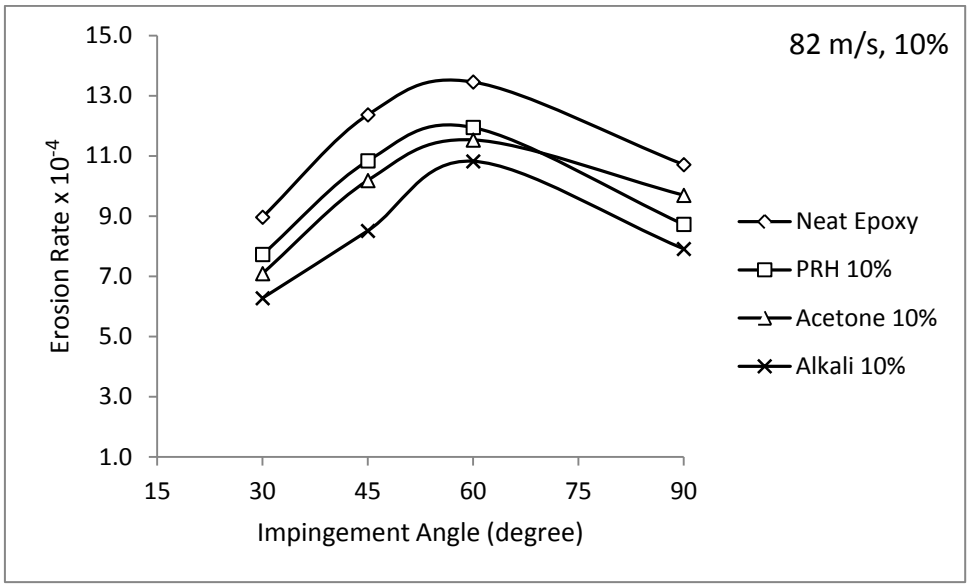


Figure-4.13: Comparison of erosion rate versus impingement angle of neat epoxy & 10% wt. fraction of PRH and treated RH epoxy composite at the velocity of 82 m/s.

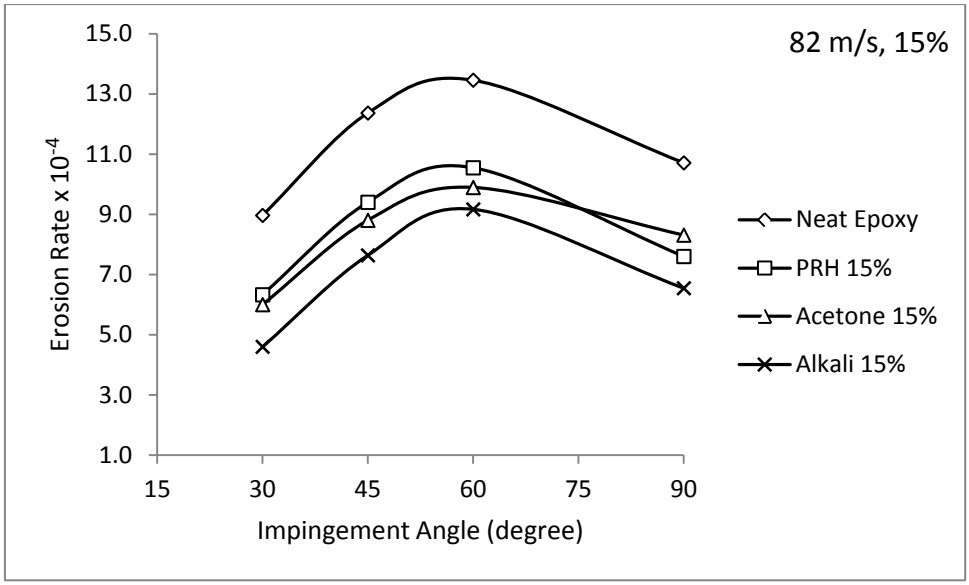


Figure-4.14: Comparison of erosion rate versus impingement angle of neat epoxy & 15% wt. fraction of PRH and treated RH epoxy composite at the velocity of 82 m/s.

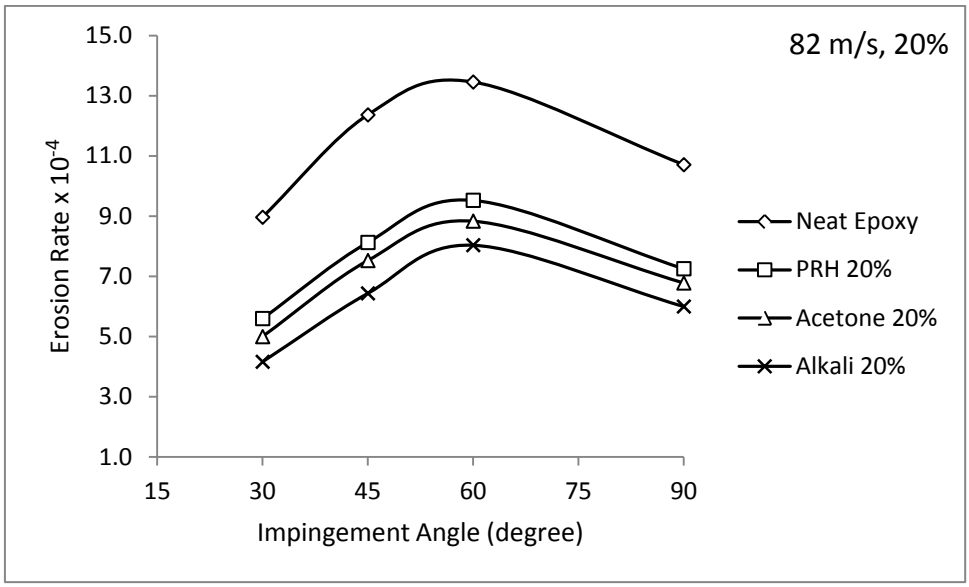


Figure-4.15: Comparison of erosion rate versus impingement angle of neat epoxy & 20% wt. fraction of PRH and treated RH epoxy composite at the velocity of 82 m/s.

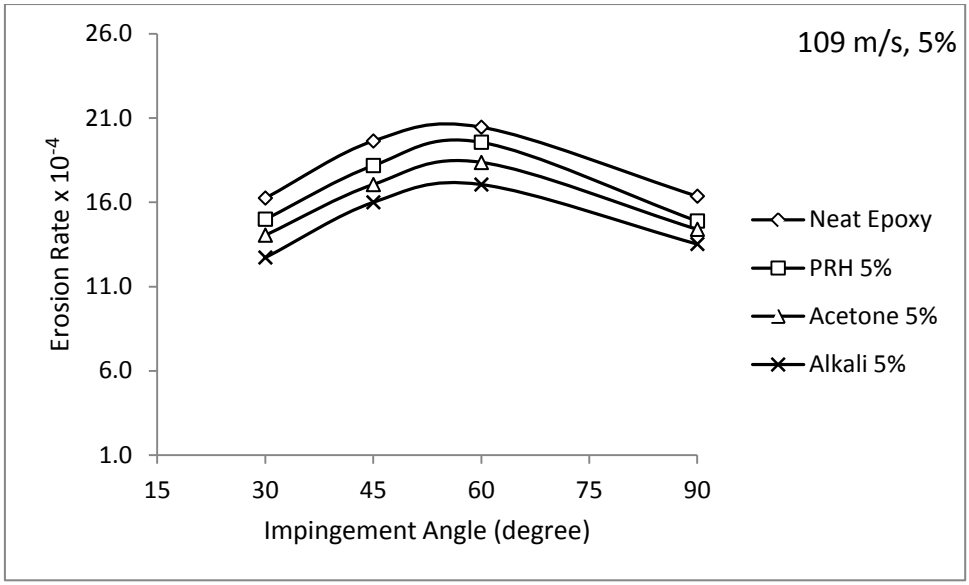


Figure-4.16: Comparison of erosion rate versus impingement angle of neat epoxy & 5% wt. fraction of PRH and treated RH epoxy composite at the velocity of 109 m/s.

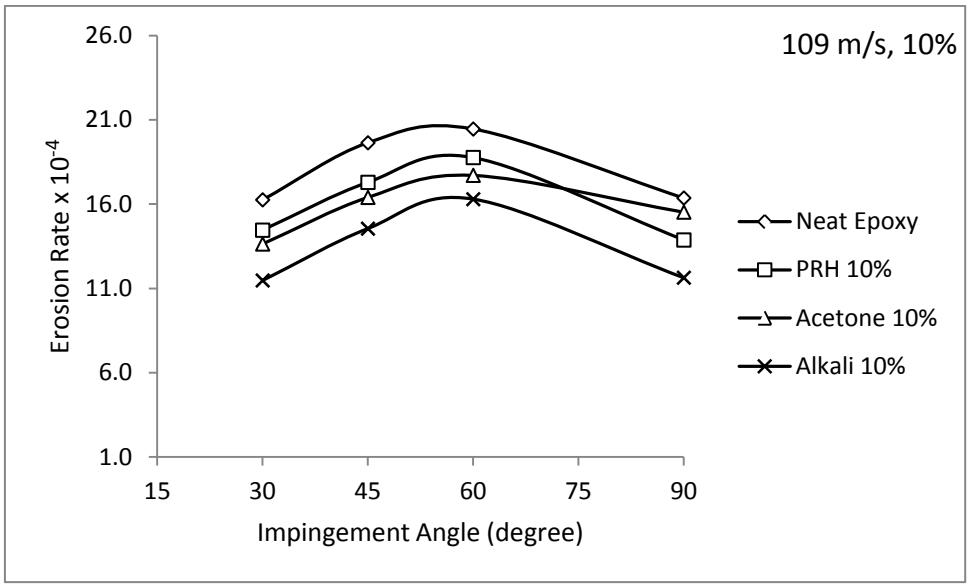


Figure-4.17: Comparison of erosion rate versus impingement angle of neat epoxy & 10% wt. fraction of PRH and treated RH epoxy composite at the velocity of 109 m/s.

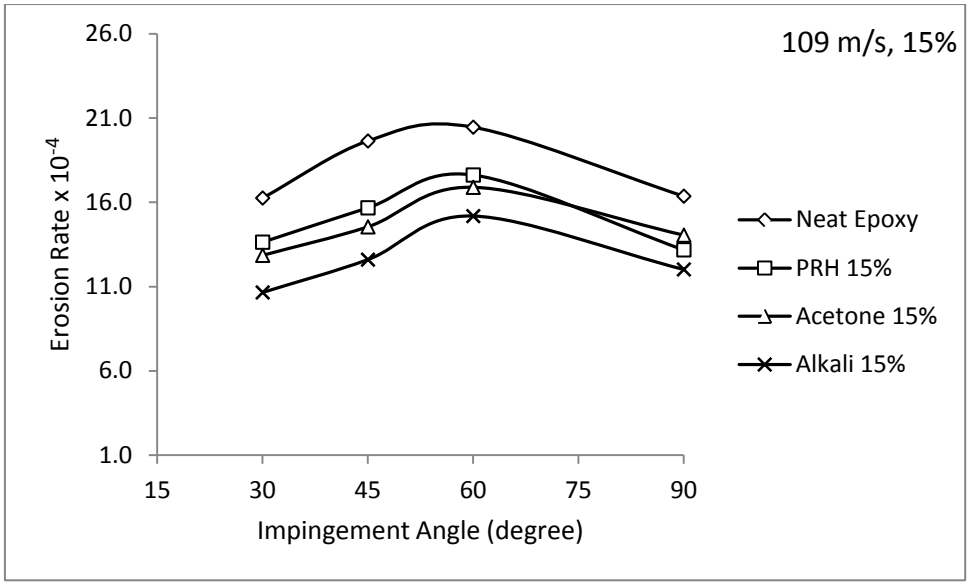


Figure-4.18: Comparison of erosion rate versus impingement angle of neat epoxy & 15% wt. fraction of PRH and treated RH epoxy composite at the velocity of 109 m/s.

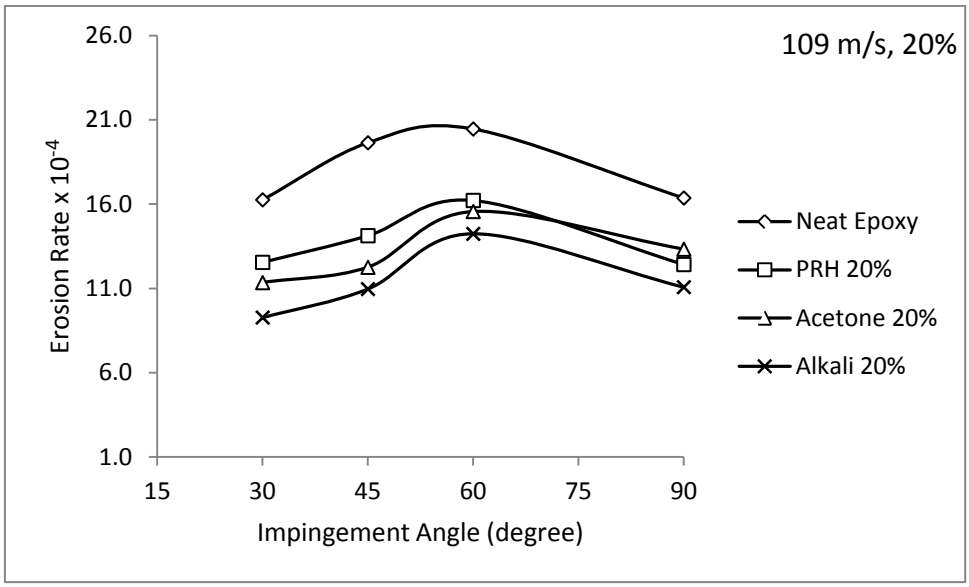


Figure-4.19: Comparison of erosion rate versus impingement angle of neat epoxy & 20% wt. fraction of PRH and treated RH epoxy composite at the velocity of 109 m/s.

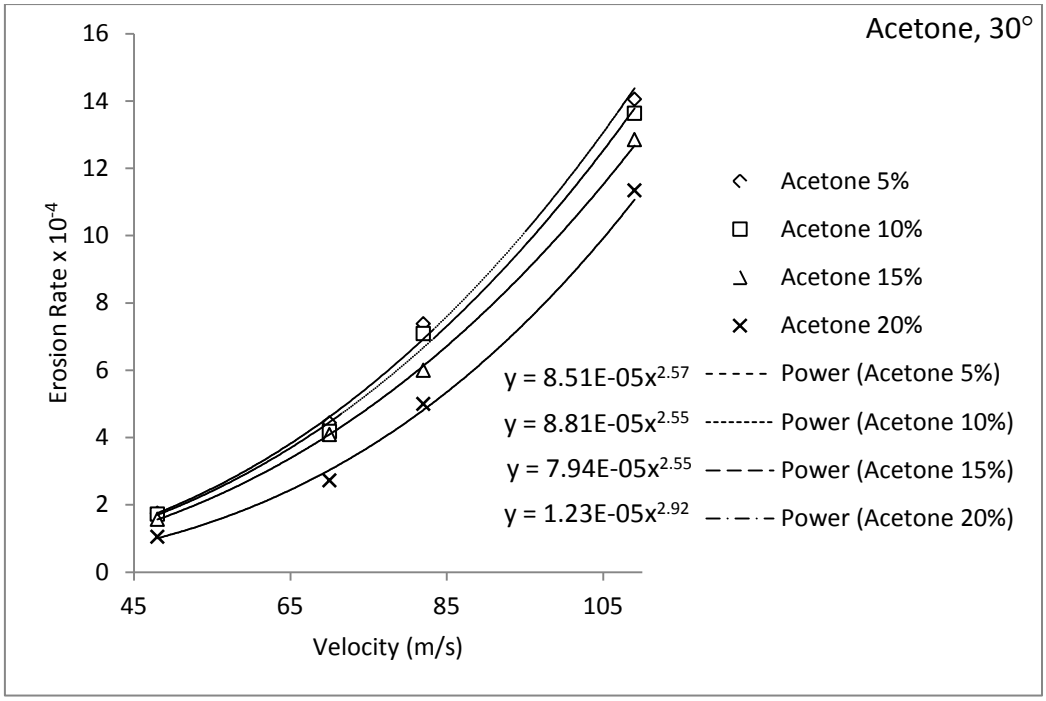


Figure-4.20: Variation of steady-state erosion rate of acetone treated RH-epoxy composites as a function of impact velocity for 30° impact angle.

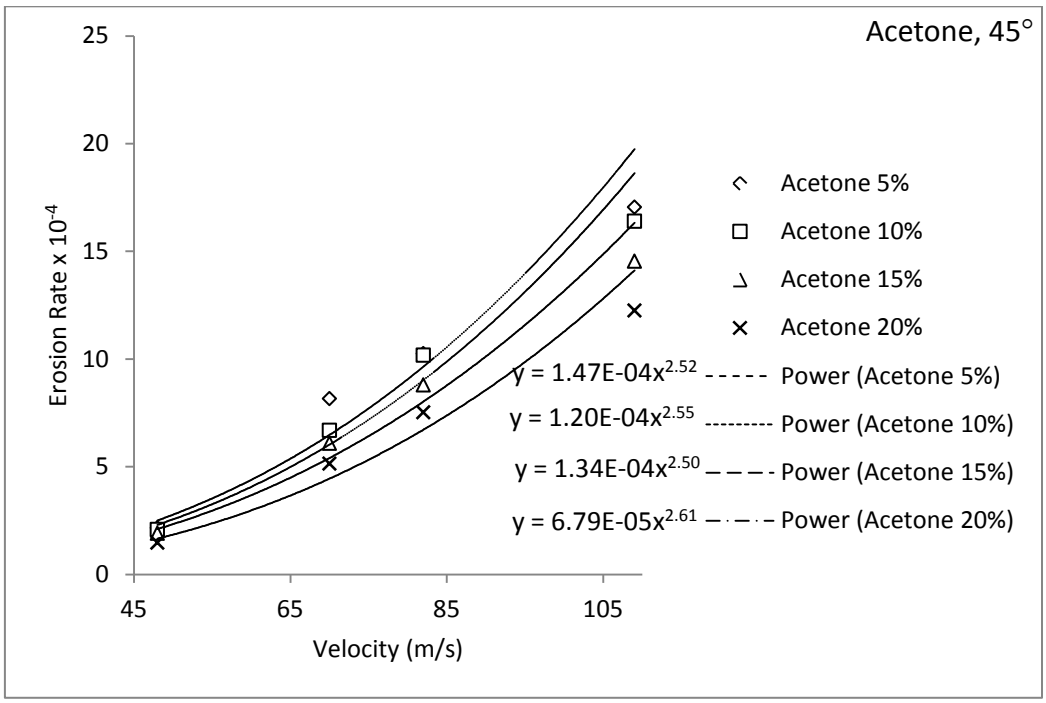


Figure-4.21: Variation of steady-state erosion rate of acetone treated RH-epoxy composites as a function of impact velocity for 45° impact angle.

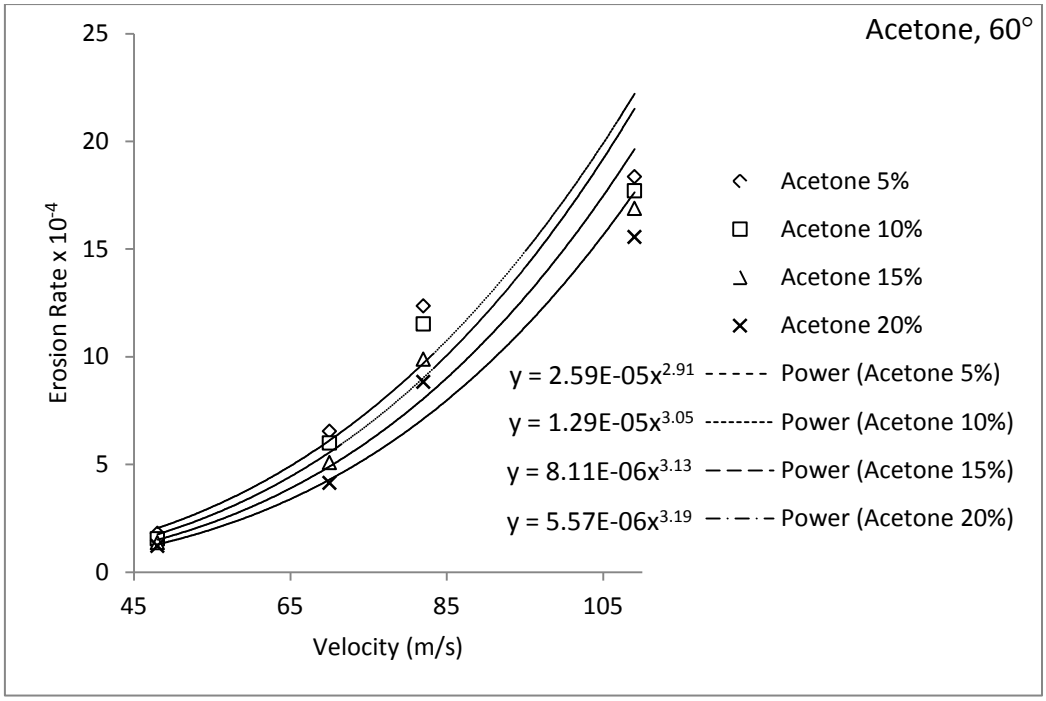


Figure-4.22: Variation of steady-state erosion rate of acetone treated RH-epoxy composites as a function of impact velocity for 60° impact angle.

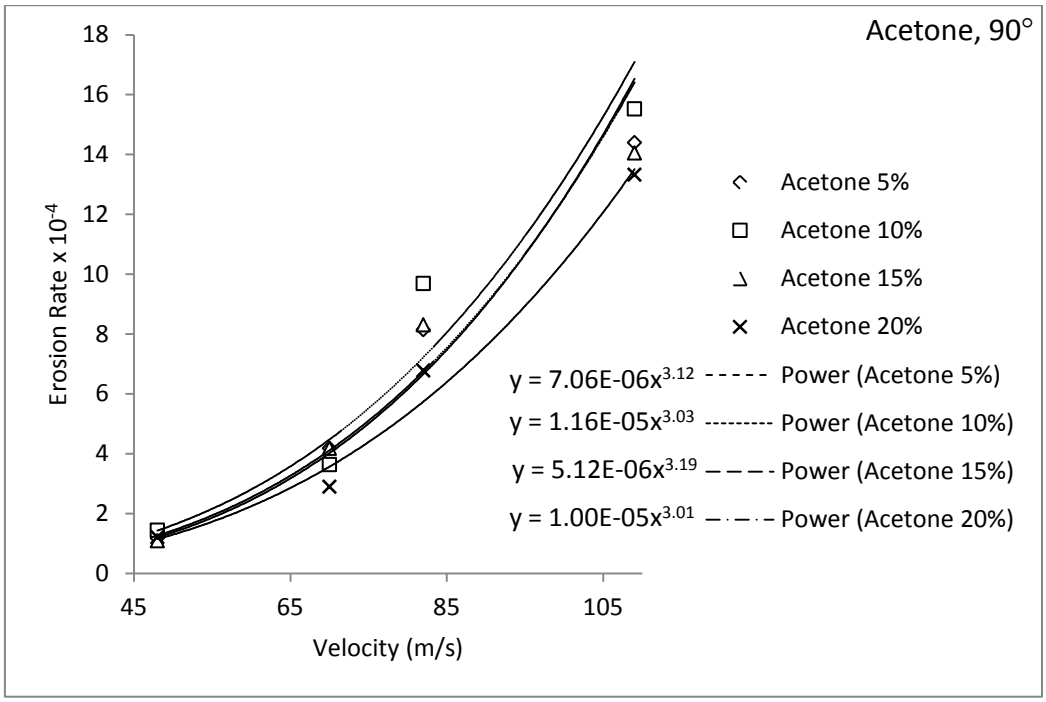


Figure-4.23: Variation of steady-state erosion rate of acetone treated RH-epoxy composites as a function of impact velocity for 90° impact angle.

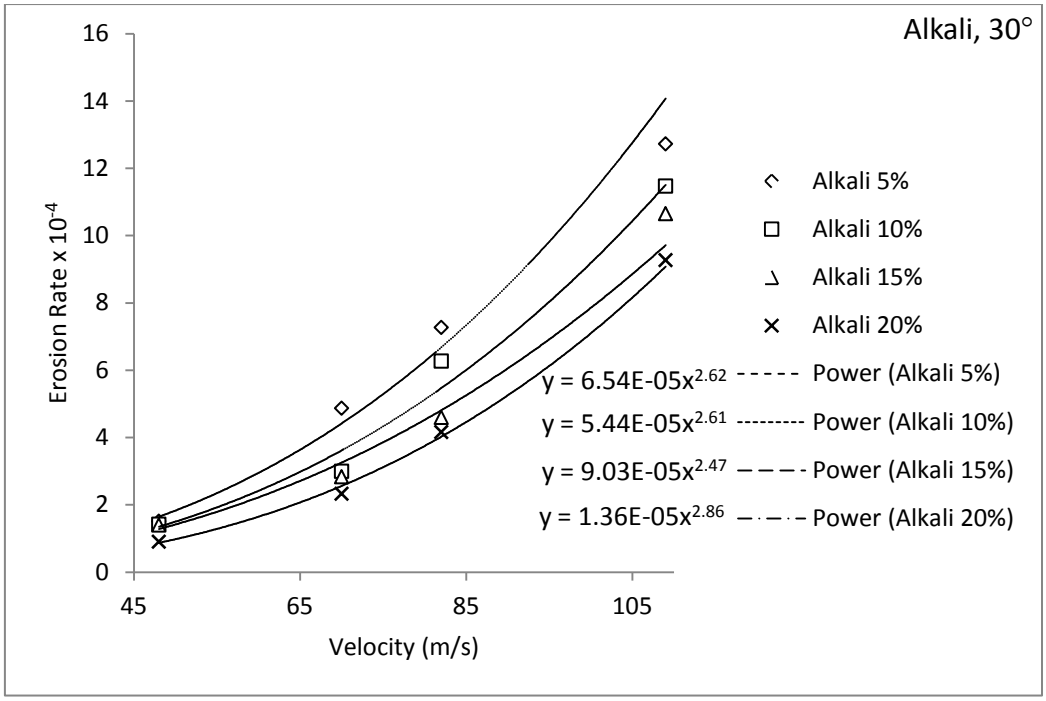


Figure-4.24: Variation of steady-state erosion rate of alkali treated RH-epoxy composites as a function of impact velocity for 30° impact angle.

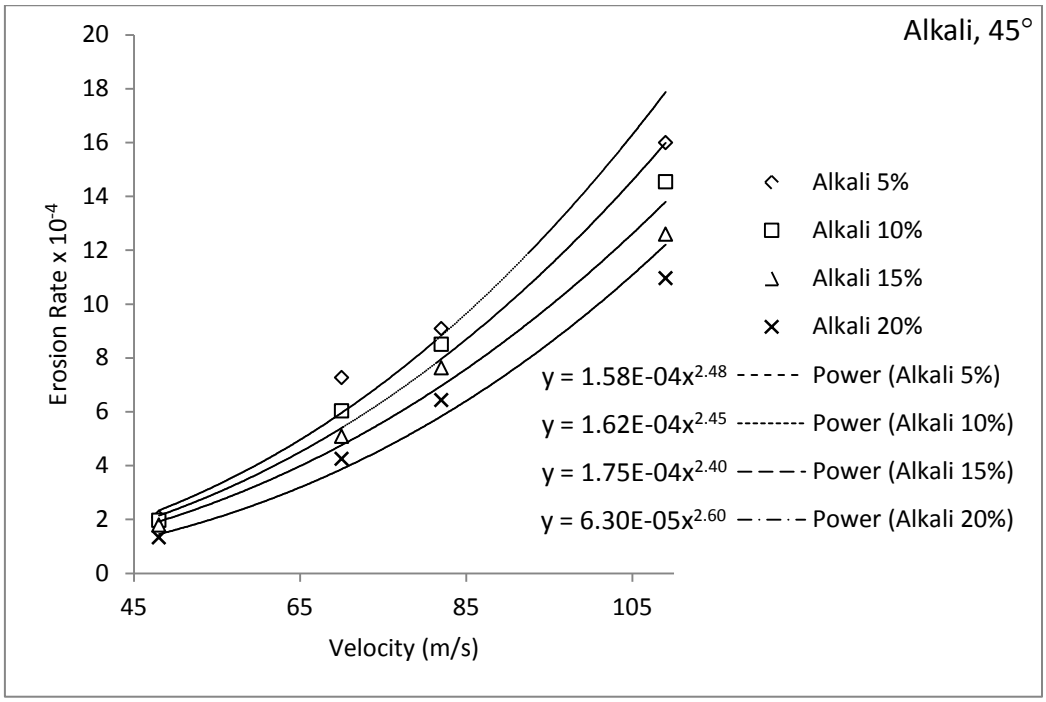


Figure-4.25: Variation of steady-state erosion rate of alkali treated RH-epoxy composites as a function of impact velocity for 45° impact angle.

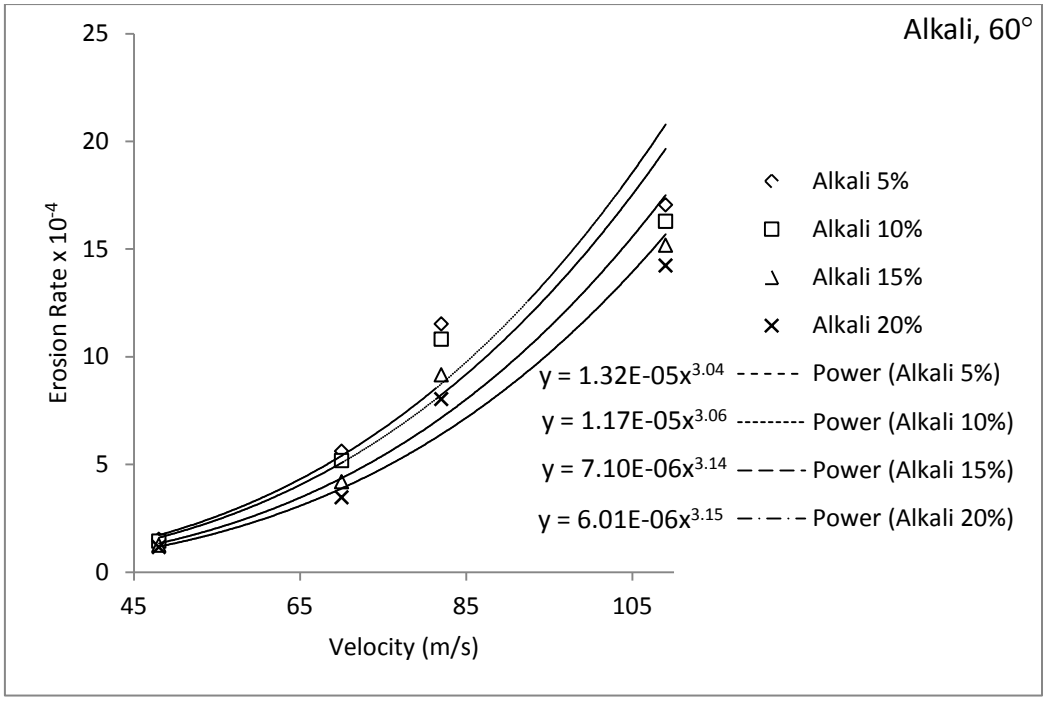


Figure-4.26: Variation of steady-state erosion rate of alkali treated RH-epoxy composites as a function of impact velocity for 60° impact angle.

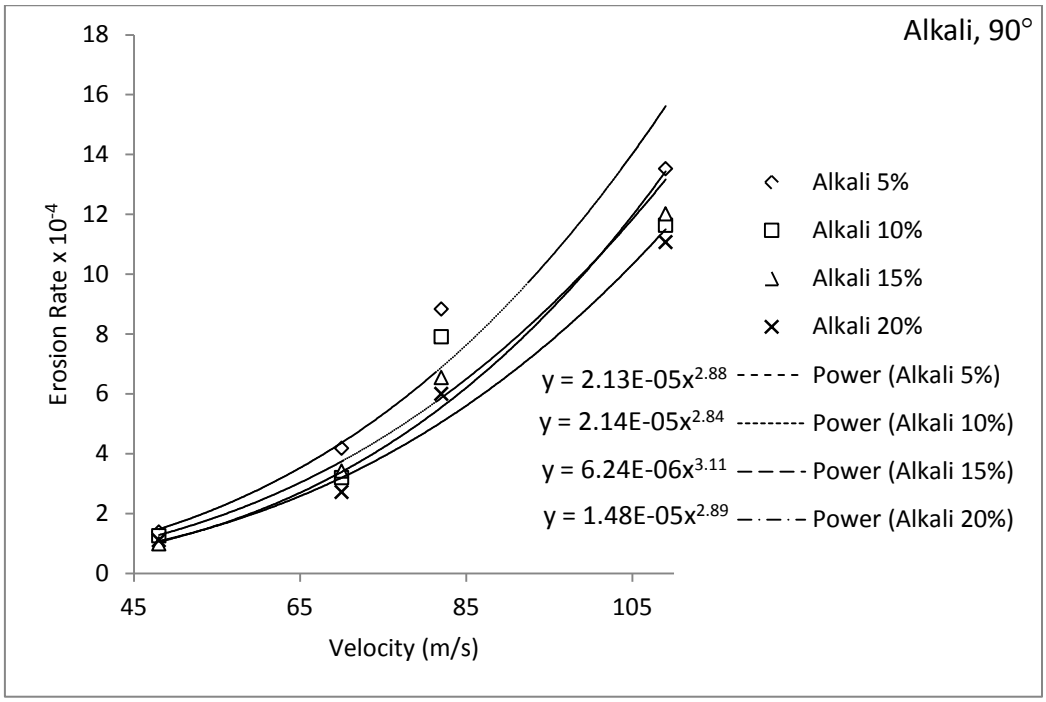


Figure-4.27: Variation of steady-state erosion rate of alkali treated RH-epoxy composites as a function of impact velocity for 45° impact angle.

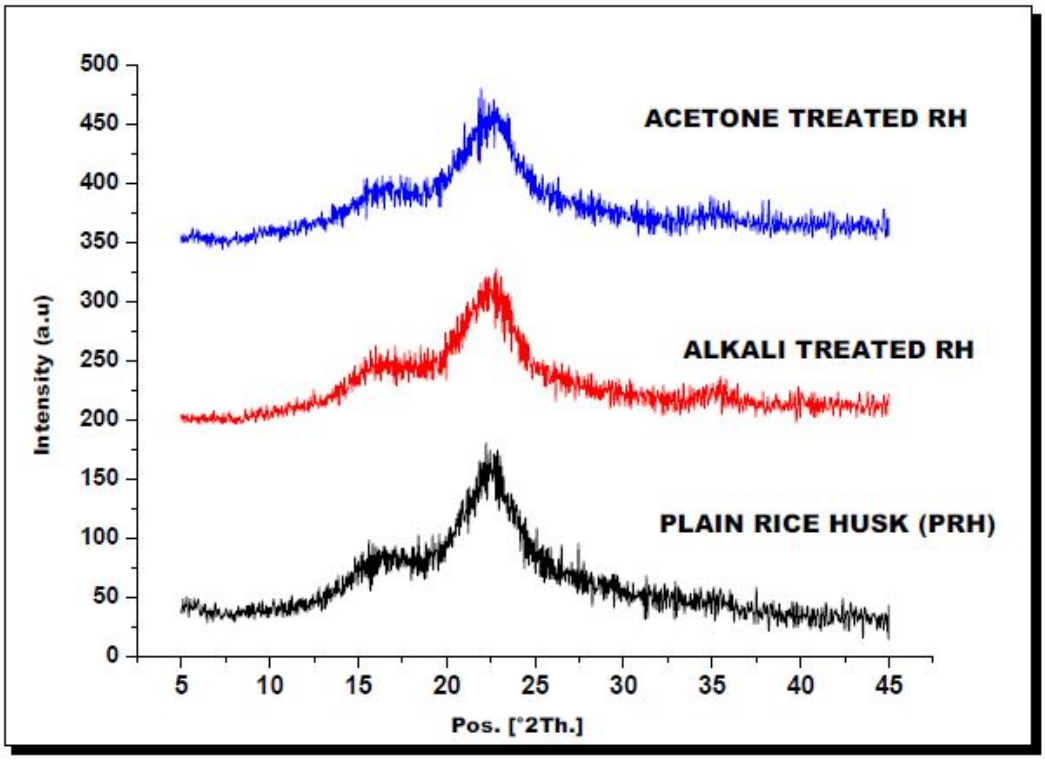


Figure- 4.28: XRD Pattern of untreated (PRH) and treated (Acetone & Alkali) Rice-Husk.

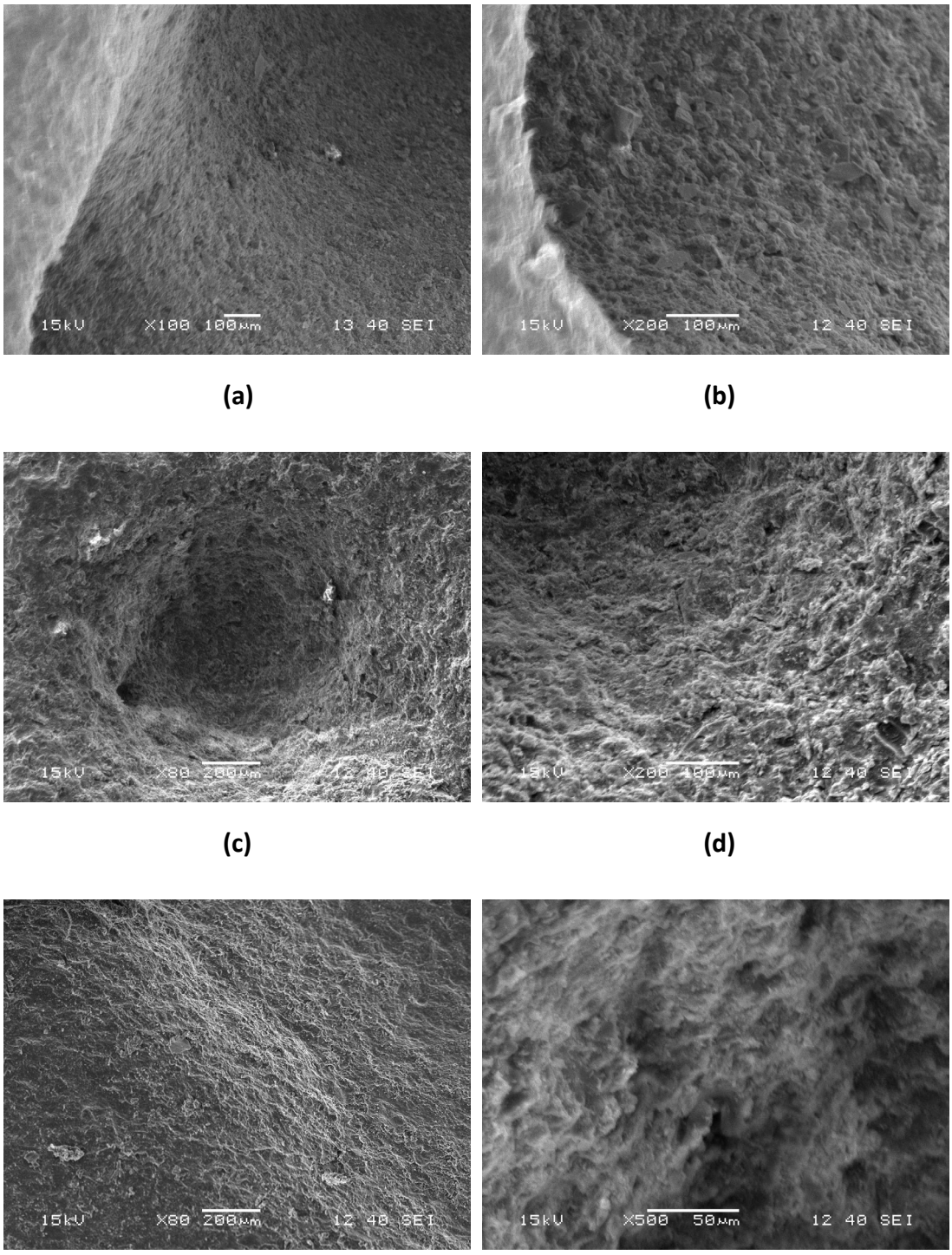


Figure- 4.29: SEM micrographs of eroded surface of (a) (b) PRH 5% (c) (d) 10% Acetone treated (e) (f) 20% Alkali treated

Chapter-5

5.1 CONCLUSION

The following conclusions are drawn from this study.

1. The Rice-Husk which is a agricultural waste product of the rice mill and cannot be decomposed easily can be successfully utilized to produce natural fiber polymer composite by suitably bonding with epoxy resin.
2. The addition of Rice-Husk fiber into neat epoxy significantly improved the mechanical and erosion behavior of the material.
3. 15% weight fraction RH fiber given the best result for tensile and bend test whereas for erosion wear test 20% weight fraction of the fiber given the best result for the composite material.
4. The RH fibers contain the silica which contributes in improving the hardness of the composite material.
5. The surface modification of fiber by chemical treatment improves the fiber matrix adhesion which further enhances the mechanical and erosive wear properties of the composite.
6. The acetone treatment provides the highest improvement in strength in comparison to alkali treatment, whereas alkali treatment given the best result for the erosion test in comparison to acetone treatment.
7. Erosion test shown that Rice-Husk epoxy composite exhibits semi brittle behavior which is conformed from the experimental result and erosion efficiency which is found to be between 1% to 6% further explains the same behavior.

5.2 RECOMMENDATIONS AND FUTURE SCOPE FOR RESEARCH WORK

1. In the present investigation a hand-lay-up technique was used to fabricate the composite. However there existing other manufacturing process for polymer matrix composite. They could be tried and analyzed, so that a final conclusion can be drawn there from. However the results provided in this thesis can act as a base for the utilization of this fiber.
2. From this work it is found that chemical modification of the fiber with acetone and benzoyl chloride significantly improves the mechanical performance of the composite. Other chemical modification methods such as silane treatment, alkali treatment, acrylation treatment isocynates treatment, Permanganate treatment, Maleated coupling agents could be tried and a final conclusion can be drawn thereafter.
3. The erodent sand particle size used for erosion test was between 300 to 600 μm whereas the same test can be conducted with different erodent particle size.

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